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A SURVEY OF POLYCHLORINATED BIPHENYLS IN AMBIENT AIR IN ONTARIO

PHASE 1

DEVELOPMENT OF LABORATORY AND FIELD PROCEDURES

FINAL REPORT

Report Number ARB-TDA-08-80

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Ministry
of the
Environment
Ontario

The Honourable
Keith C. Norton, Q.C.,
Minister

Graham W. S. Scott, Q.C.,
Deputy Minister

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PHASE I

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PREPARED BY
MONITEQ LTD.
630 RIVERMEDE RD.
CONCORD, ONT.

AND

NUCRO TECHNICS
2000 ELLESMERE RD.
SCARBOROUGH, ONT.

AND

ONTARIO MINISTRY OF ENVIRONMENT
AIR RESOURCES BRANCH
880 BAY ST., TORONTO

JANUARY, 1981

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This report is part of a series of reports published by the Ministry of the Environment, Air Resources Branch - on the development of techniques for collection, detection and determination of Polychlorinated Biphenyls in ambient air and results of surveys performed in Ontario in the periods of Sept.-October 1979 and June 1981. The reports of this series are:

ARB-TDA-08-80-Phase I - Development of Laboratory and Field Procedures

ARB-011-81-ARSP-Phase II - Volume I; Sampling Site Selection and Analytical Procedures.

ARB-025-81-ARSP-Phase II - Volume II; Meteorological Correlation for the 1979 Survey of Polychlorinated Biphenyls in Air in Ontario.

ARB-026-81-ARSP-Phase III - 1980 Survey of Polychlorinated Biphenyls in Air in Ontario.

This series of reports should be considered as a unit, and the individual members of the series should be evaluated in concert with the other members. This situation arises since this work was developmental in nature and the reports present a chronological picture of the work done over period of time on the sampling, sample processing and analytical procedures. The Phase III Report describes the current state-of-the-art for PCB sampling and analysis as developed by MOE Scientists.

ES/mb/AR2-38

INTRODUCTION

The information in this report provides a summary of the results of the development work which has been undertaken in relation to the analysis of polychlorinated biphenyls in the ambient air. Prior to undertaking the analysis of actual samples, it was essential to ensure that all procedures utilized for the analysis were well characterized and worked well in our hands. It is not uncommon in the field of trace analysis that techniques which work and produce results for one group of people will not work for others. To this end, we have endeavoured to provide a detailed protocol for all aspects of the analytical program, which serves as a working manual, and hopefully, will permit others to successfully apply this methodology in the future.

Gas chromatography utilizing electron capture detectors was the method chosen for sample analysis. This was dictated by the complexity of organic volatiles contained in real air samples and the very low atmospheric concentrations of the PCB isomers. The use of isomer standards for the analysis has many advantages (which are detailed later); the greatest advantage being a better understanding of the problems associated with their analysis in environmental air samples.

A clean up procedure can only simplify the quantitation of the final extract and, if done properly, improve the accuracy by eliminating interfering components. The clean up method, using "Florisil", is a routine procedure used in all areas where PCB's and pesticides are monitored. We have confirmed its suitability in our case, and hopefully, improved the reliability of the technique.

Pentane solvent is being used for all procedures at present with many advantages; higher recoveries during solvent evaporations being the main justification. But the use of pentane alone, means that larger numbers of samples can be processed daily, making a province-wide air survey feasible in both a practical and economic sense.

The technique used for air sampling in the field has been used extensively by Ministry of Environment Lab Services Branch for some period of time and was used as recommended without any changes. (1)

INSTRUMENTATION AND ANALYTICAL CONDITIONS

1) Gas Chromatography - Column Selection
Various liquid phases were evaluated for their suitability for the analysis of PCB's. The very low levels of PCB's to be monitored, the complexity of PCB samples in general, and the need to use temperature programming to permit analyses in a reasonable length of time dictated that the column meet the following criteria:

i) Adequate resolution of PCB isomers
ii) Minimal baseline drift
iii) Good reproducibility (short and long term)
iv) Reasonable column longevity.

Three liquid phases were briefly investigated. "Dexsil 400", "Dexsil 410" and "OV-17" were tested as 3% loadings on "Chromosorb WHP" 80/100 Mesh. "Dexsil 400" was chosen for use because of the reduced column bleed which was evident and efficiency which was comparable to the more commonly used "OV-17". "Dexsil 410" offered no advantage and resulted in somewhat longer analytical times due to the higher polarity. The degree of baseline drift during temperature programming was a significant criterion as well. "Dexsil 400" liquid phase also exhibits less drift during temperature programming than other materials used; can be used at low loadings (1%) without significant effect on column reproducibility and longevity, and produces columns which are as efficient as other more commonly used materials.

The "Dexsil 400" column described was used for an extensive period for evaluation of other aspects of the methodology even though it was later realized that the resolution and total efficiency would be inadequate for analyses of ambient air extracts.

Resolution was improved by utilizing a 12 ft. column and lowering the loading of "Dexsil 400" liquid phase from 3% to 1%. Since it was felt that actual air samples may require a short higher temperature cycle at the end of the analysis to prepare the system for the next sample, two non-silanized solid supports were tested; "Dexsil 400" not being recommended for use on a silanized support at high temperature. "Anakrom A" 90/100 mesh was used for the additional advantage of increased performance due to the narrower mesh cut. Ultrabond II was used on the recommendation of G.A.V. Rees (MOE-LSB), because of the improved characteristics of coating, and improved resolution. This latter support material produced the most efficient columns with a 1% "Dexsil 400" loading of any that were tested, but we were unable to produce a column with the necessary stability and longevity. This is in contrast to G.A.V. Rees who has been using this material with a marked degree of success. It was suspected that the difference may be due to the presence of trace oxygen contamination in the different carrier gases in use (Nitrogen vs. Argon/CH_4), although the problem was not eliminated with the incorporation of a commercial oxygen trap. "Anakrom A" (80/90 Mesh) was finally used, with a 1% "Dexsil 400" loading. This column is as efficient as the previous one, but exhibits some differences in selectivity.

The following are the conditions used for the analysis of PCB's:

Column - Glass Coil 3.6 m. x 2 mm I.D. x 6 mm O.D.
Packing - 1% Dexsil 400 on "Anakrom A" 80/90 Mesh
Injector Temperature - 190°C
Detector Temperature - 260°C
Column Temperature - 160°C - 4 min hold
    Program - 1°C/min - 33 min
    Program - 4°C/min Hold at 220°C
Carrier Gas/Flowrate - Ultra Pure Nitrogen conditioned with an "Oxisorb" and Molecular Sieve 5A trap, 17.5 ml/min (for Varian 3700 only)
Carrier Gas/Flowrate - 5% Methane/Argon conditioned with a Matheson 8301 "Hydrox Purifier" and a Molecular Sieve 5A trap, 19.5 ml/min (for HP Model 5740 only)

2) Instrumentation

a) A Varian 3700 gas chromatograph fitted with dual Ni^{63} electron capture detectors and a Varian Model 8000 auto injector. Digital integration of the detector outputs and partial data reduction are provided by two Varian CDS-111 Chromatography Data Systems. This comprises the main analytical system used for the analysis of PCB sample extracts.

b) A Hewlett Packard 5740 gas chromatograph fitted with a Ni^{63} electron capture detector. A Hewlett Packard 3380 A Reporting Integrator provides the digital output and recorder trace. This comprises a back-up system which is used for monitoring of glassware provings and other ancillary analyses.

3) Linearity and Precision of Assay

The accuracy of the final analysis is very much dependent on the linearity of the response of the detector signal, integrator system, and the precision of the analysis. Linearity and precision of assay are two parameters which are being monitored on a continual basis to ensure the accuracy of analysis and to permit continual confidence in the final instrumental assay.

For reasons of time and economy it was necessary to utilize a single point calibration of the analytical systems. Multipoint calibration would have required complete manual data reduction, since the CDS-111 data reduction systems are capable of handling only single point calibration. Single point calibration can cause significant errors in analysis if the actual linear calibration exhibits a significant intercept value.

The linearity of response to different concentrations of a multicomponent standard was used as the test which ensured that the integrator was programmed correctly. Incorrect setup would produce peak area ratios which differ significantly from the relative dilution factor of the standards used for the test. The Varian CDS-111 systems which are used for integration of the detector signal and data reduction allow control of parameters which affect the integrator output. In general it was found best to set the signal to noise parameter so that average baseline noise was not detected; set the peak width parameters to compensate for the increasing peak width during temperature program; and incorporate appropriate instantaneous forced baselines between standard peaks to ensure that the baseline is tracked during temperature programming.

The final program listing, whereby acceptable linearity was achieved, is included in Table 1. The results of the final study in which a multicomponent standard was analyzed at two levels, indicate an acceptable level of linearity in the range of interest (Table 2).

The deviations which are evident in the results of one-hundred-fold dilution are due to the detector signals being near or below the detection capability of the integrator, resulting in either missing the peak completely or in late detection. It is at this low level (14.9 ng PCB's/ml) corresponding to 1.5 ng PCB's per cubic meter of air that the analytical error is significant. Component peaks of this size are extremely small and are handled manually if required.

The precision of the chromatographic assay was determined using both manual injections (incorporating the solvent flush technique) and utilizing the autoinjector system (Varian Model 8000). Results of repeated manual injections are given in Tables 3 and 4. Retention times are reproducible; the variation ranges from 0.18% to 1.99%.

The last figure of 1.99% which is high in comparison to the rest is due to faulty electronic circuitry which permitted a minor temperature overshoot during the final hold in the temperature programme. Area determinations are less precise but are considered adequate for the job. Variations ranged from a minimum of 2.0% to 30.7%.

Precision of assay was more difficult to achieve utilizing the auto sampler/injector system. Initial set up and our lack of familiarity with the particular system (a Varian Model 8000) were contributing factors. This system does not utilize the injection syringe itself to extract a sample aliquot from a vial, but air pressurization of the sample vial to positively displace the sample via a "Teflon" transfer line into a side port filling the injection syringe. The protocol for sample extraction and clean up produced originally a final extract in 0.5 ml of iso-octane. The sample vials used, have a capacity of 2 mls. The manufacturer recommends that these vials be at least ½ full, and that a volume of 250 ul be transferred through the line for reliable operation. Attempts were made to utilize smaller sample volume and smaller purge volumes, since we were not willing to utilize 50% of our sample for purging the lines. It was ultimately realized that sufficient variation in the shape of the vials existed, that the sample exit port in the needle entering the vials would be exposed to the pressurized air as the liquid level dropped. Reducing the purge volume (effected by reducing the level of pressurization of the vial)down to 150 ul proved more reliable; below that level, pressurization was insufficient to consistently drive the sample through the line. In addition problems were encountered with pieces of septa plugging the sample exit port of the needle which pierces the vials. This problem was traced to a burr on the tip of this needle which was tearing the septa. (It was later realized that this itself may have caused the inconsistent results we observed, by permitting leakage of the pressurization air, affecting sample transfer).

Claims by the manufacturer that smaller flush volumes (50 ul) could be used if micro vials were utilized; were not substantiated even though we purchased a quantity of these vials. Since we were able to obtain reproducible results with a purge volume of 150 ul; finally a sample volume of 1.0 ml was used to minimize the percentage of total sample used and eliminate the possibility that the sample level would fall below the exit port.

A possible source of cross contamination of samples is the adsorption of PCB's onto the surfaces which contact the sample, i.e. the "Teflon" line, stainless steel needles and fittings and the glass barrelled injection syringe. To eliminate this possibility, the system was set up to wash the transfer lines with blank solvent (to eliminate all residuals) immediately after injection of a sample. This is not the standard mode of operation for this system and requires a slight modification which precludes the possibility of multiple injections from a single sample vial.

The reproducibility of the autosampler/injector was demonstrated by repeated injections of a standard contained in vials each separated by a wash vial. The efficacy of the wash was proven by injection of a blank solvent after a wash. No visible peaks were evident in the chromatogram of the blank (Fig. 1) which corresponded to components in the previously run standard (Fig. 2). Reproducibility of retention times ranged from 0.72% to 2.32% while area determinations were reproducible from 0.66% to 6.93%. Details are given in Tables 5 and 6.

Comparison of data between manual and automated injections does not indicate that there is significant gain in precision when automation with the Varian Mod. 8000 sampler is used. Careful manual injection can provide results as consistent as an automated system. The automated system, however, offers the convenience of unattended operation especially overnight.

4) Detection Limits

The detection limits of isomers using the Varian 3700 system have been determined. For all practical purposes the Varian CDS-111 will not detect and itegrate peaks less than one thousand area counts (arbitrary units). These limits (in pg/inj. volume) are listed in Table 7. Also included in this table are the absolute detection limits based on a manual interpretation of peak size relative to detector noise level. Detection limit being defined as that quantity which produces a detector response twice the noise level (peak to peak).

5) System Stability

One major factor which is important for accuracy of assay is the stability of the analytical system. Since the mass response of the electron capture detector increases strongly as additional chlorine atoms are added to the ring system and even varies among geometrical isomers (with the same number of chlorine atoms), accurate analysis depends on correct identification of the isomer peaks and assignment to a corresponding peak in the calibration mixture. This would not be feasible if sufficient stability had not been achieved. The quality control protocol (Appendix A) which has been developed for this program demands that calibration of all systems is done routinely and consistently. Retention times of all isomers in the standard, and the area responses are tabulated and viewed over the long term. The results of such monitoring are given in Table 8. This table indicates the level of stability that has been achieved over a period of seven days.

EQUIPMENT, GLASSWARE AND REAGENT PREPARATION

Due to the complexity of the analysis and the very real possibility that inadvertant contamination will significantly affect the accuracy of the analysis for PCB's; a great deal of effort was expended in this area to ensure that all equipment, glassware and reagents used are as clean as possible on a consistent basis. All details of the cleaning, proving and maintenance of clean glassware are given in the overall analytical protocol, Appendix A.

1) Equipment

Ovens are used for three purposes in the analysis of PCB's:

i) High temperature (250°C - forced air) ovens are used for baking of solvent rinsed glassware to vaporize organic residues.

ii) Low temperature (135°C - convection) ovens are used for reactivation and storage of sampling cartridges and for storage of "Florisil" used for sample clean-up.

iii) Low temperature (135°C - convection) ovens (separate from above) are used for storage of glassware which has been solvent rinsed immediately before usage and is awaiting proof of its cleanliness.

NOTE: It was later realized during large scale production of sample extract and clean ups; that the large amount of glassware used takes considerable time to heat up and to cool down. It was necessary to wait until the glassware was cooler than 36°C (boiling point of pentane) before the glassware was rinsed with solvent. Consequently, to maintain a reasonable level of productivity and to avoid anaesthetizing the technicians, glassware was left at room temperature (no longer than required for proof of cleanliness) before use. No overall change in subsequent blank values was observed.

All of the ovens which are in use had been cleaned prior to usage in the following manner:

- a thorough cleaning with soap and water to eliminate dust
- a rinsing with water
- bakeout at maximum temperature until no odour and smoke was detected
- complete rinse of all interior components with acetone followed by pentane.

The suitability of the ovens in use were proven by storage of materials within them and subsequent proving of the glassware. Twelve sets of glassware were baked and proven clean and restored in the high temperature oven. Two sets were removed, twice daily over a three day period, allowed to cool to room temperature and rinsed with pentane. The pentane rinsing from the two sets was concentrated in the first set. The second set of the two was rerinsed with pentane and concentrated. Any contamination of the glassware which would occur in the oven would be removed by a pentane rinsing. The rerinsing of the second set would permit a comparison with the residual level due to solvent. No significant difference in contaminant levels was noticed in this experiment during the 3 days that the glassware was tested. Expanded aluminum mesh baskets are used for storage of glassware while baking in ovens. These were cleaned and proven in an identical manner to the ovens previously described. During the course of this phase of the contract a severe contamination problem occurred which took some time to eliminate. Gross contamination of the glassware was evident in the glassware provings. Repeated rinsing with solvent and further baking in the high temperature ovens was to no avail. The contamination was ultimately traced to plastic gloves which were used to minimize contact of the glassware with bare hands. It had become apparent that one (or more) of our personnel had inadvertantly handled one of the expanded metal mesh baskets while hot, melting the glove onto the metal. Without realizing the significance, this basket eventually returned to the high temperature oven where the plastic (polystyrene) would decompose and deposit organic vapour onto the glassware. After cleaning of this contaminated basket with steel wool and solvent rinsings, the contamination level subsided. Cotton gloves, which are thoroughly cleaned with solvents and proven are now used for handling of the glassware, eliminating the possibility of any skin oils or plastic contacting the outer surface of the glassware and vapourizing onto the inner surfaces which contact sample extracts. A useful procedure developed for tracing contamination on oven walls, or other surfaces suspected of being sources of contamination is to hold a clean cotton ball in a pair of forceps; saturate with pentane and "swab" the surface picking up contaminant materials. The cotton ball is subsequently squeezed to release the solvent which can be assayed by GLC to observe any characteristic fingerprint.

2) Glassware

All glassware and metalware which is used for the sampling, sample extraction and work-up or sample storage undergoes extensive cleaning and proof of its suitability for use before it is actually used. The procedure, which is detailed in Appendix A, involves initially a thorough soaking in 2% "Decon" (a non-ionic surfactant), followed by a thorough wash with distilled water and rinse with acetone, then pentane. The glassware is allowed to drain of solvent in the metal baskets and is subsequently baked at 250°C overnight.

Glassware is handled in batch sizes corresponding to glassware required for the extraction and preparation of ten samples. After an overnight bakeout, the glassware is cooled to room temperature and all surfaces which will contact the sample extract are rinsed with pentane. The rinsings are collected and concentrated to 1.0 ml (iso-octane) and the concentrate subsequently analyzed by GC. This analysis is the basis on which the cleanliness of the glassware and its suitability for use with field samples is determined.

Glassware, suitable for use, has a proving analysis which is equivalent to an ambient air analysis of one ng/m^3 or less of total PCB's.

3) **Reagents**

i) Solvents - All solvents used were grades designated by the supplier as suitable for pesticides residue analysis. Solvents were proven prior to use by GC analysis. An acceptable analysis was equivalent to an ambient air analysis of one ng/m^3 or less of total PCB's. Solvents that did not meet this criterion were rejected.

ii) "Florisil" - Two mesh sizes of "Florisil" were used:

a) "Florisil" - Standard Grade 30/60 mesh (used as the collection absorbent in the sampling cartridges), was thermally cleaned and proven before use as indicated in the detailed procedures. (Appendix A)

b) "Florisil" - Standard Grade 100/200 mesh (used for column chromatographic clean-up of ambient air extracts), was thermally cleaned, and deactivated with 3% H_2O by weight and slowly tumbled for twenty-four (24) hours before use.

iii) PCB isomer standard mixtures were prepared from the stock standard solutions supplied by Air Resources Branch of the Ministry of Environment.

The "Florisil" as supplied by "Floridin Comp." was virtually free of contaminants which would coeluete with PCB's. This was likely due to a change in packaging by the manufacturer. The large fibrepac bulk containers were lined with an unplasticized "Mylar" bag containing the "Florisil" as opposed to the more flexible plastic pouch previously used. Contaminants which were present were of low molecular weight and high volatility. A brief comparison of continuous solvent extraction and the thermal clean up (tube furnace) indicated that the thermal clean up produced a material in which no quantifiable contaminants remained. After solvent extraction, some new contaminants were observed which were previously not present (See Figures 3, 4, & 5).

All "Florisil" was thermally precleaned prior to use. A quartz tube (69 mm O.D. x 65 mm I.D.) was loaded with "Florisil" (400 gm), held in place with a glass wool plug. The quartz tube was mounted vertically (to prevent channeling of the air stream) in a tube furnace and heated slowly to 650°C, while a slow stream of "Florisil" filtered air (1.1/min) passed through the bed. The "Florisil" was maintained at 650°C for twenty-four (24) hours, then cooled to room temperature for use (described in more detail elsewhere in this report and Appendix A).

Thermal precleaning has significant advantages over solvent extraction:

a) Ability to handle large quantities in relatively short time
b) "Florisil" is reactivated as it is cleaned. Solvent extraction requires that the material be reactivated afterwards.
c) A one-step process - thorough cleaning by solvent extraction requires that two solvent systems be used in sequence; (methylene chloride, then hexane).
d) Efficiency - all organics are removed either through desorption and vaporization or ultimately by oxidation at the high temperature used in the presence of air.
e) Safety - no need to handle large volumes of solvent, or manipulate large quantities of solvent laden "Florisil" which must be dried and reactivated.

Prior to use, each batch of thermally cleaned "Florisil" was proven suitable for use, (as described in Appendix A), and had a proving analysis which was equivalent to an ambient air analysis of one ng/m^3 or less of total PCB's.

4) Sampling Cartridges

Although all components which go into the production of the sampling cartridges were proven on an individual basis, it was felt that there was sufficient risk of contamination during the process of assembly to incorporate further testing after assembly and prior to shipment into the field.

After assembly, cartridges were eluted with two solvent systems:

i) 200 ml 10% CH_2Cl_2/pentane
ii) 200 ml pentane.

The last 30 mls of the pentane eluate from ten cartridges were combined. The combined extract was evaporated to 1.0 ml (iso-octane) and analyzed by GC. Residual pentane on the cartridges was removed by passing of slow stream of "Florisil" filtered air through the cartridges, until the odor of hydrocarbon vapor was no longer evident. The cartridges were subsequently reactivated at 135°C and accepted if the proving assay was equivalent to an ambient air analysis of one ng/m^3 or less of total PCB's. No attempt was made to reclean or reclaim cartridges if they did not meet this criterion. The removal of the pentane is important to eliminate the potential fire hazard when the cartridges are placed in the oven. Secondly, small amounts of pentane remaining on the "Florisil", not necessarily being a fire hazard, will produce a strong pungent odor when the cartridges are in the oven. This odor (also a strong lachrymator) may result from the oxidation of residual pentane and may be valeraldehyde, formaldehyde or formic acid. It is essential that all residual pentane be removed before placing cartridges in the oven. These same cartridges which produce an odor would eventually discolor if left in the oven, the "Florisil" becoming a pale tan brown. Other than the discoloration there was no indication that these cartridges were contaminated (GC assay). Full reactivation of cartridges required a storage of two days at 135°C.

Prior to shipment to the field, one cartridge from each batch of 10 was selected and analyzed to ensure no contamination had occurred during the period of reactivation and storage. (Refer to Appendix A for details). The group of nine remaining cartridges were accepted for shipment to a field sampling site if the blank proving assay was equivalent to an ambient air analysis of one ng/m^3 or less of total PCB's.

DEVELOPMENT OF SAMPLE PROCESSING PROCEDURES

These procedures which are currently being used for the extraction and clean-up of the PCB samples were developed over a period of time during which all operational aspects were scrutinized, difficulties evaluated and resolved.

1) Sample Recovery

The results of the preaward check samples indicated advantages in using pentane rather than hexane as elution solvent. These initial test samples in hexane took between 4 to 6 hours to concentrate from 200 ml as supplied to 0.5 ml in iso-octane used for analysis. In addition losses of the lower chlorinated isomers were significant (2). Pentane has been used throughout this study because of rapid evaporation of large volumes and the quantitative recoveries of the lower chlorinated PCB isomers.

"Kuderna-Danish" evaporators are being used because of the relative ease with which large numbers of samples can be handled, and their ability to evaporate solvent and yet allow quantitative recovery of PCB isomers. This latter point is largely due to the refluxing of the solvent within the "Snyder" column. Recovery of PCB isomers was tested with three different multicomponent standard solutions which covered the range of isomers of interest. An aliquot of each was spiked into 200 ml of pentane and recovered by evaporative concentration into 0.5 ml of iso-octane. These solutions were compared directly by GC analyses, with the same standard diluted to the same analytical concentration. The results are given in Table 9.

Recovery of PCB isomers from field sampling cartridges was tested in an equally straightforward manner (Table 10). Cartridges which had been prepared as described briefly in the text (refer to Appendix A for details), were spiked with a known solution of PCB isomers, then dried with an air stream. The PCB's were recovered by elution with pentane (200 mls), and evaporated as previously described. An experiment simulating field conditions by deactivation of PCB spiked test cartridges is described in the next section covering "Florisil" clean up of sample extracts. Sampling cartridges used in the field would absorb a large quantity of water from the atmosphere and would become deactivated. We were unable to detect any significant effect of deactivation on our recovery of PCB's. We continued to use fully activated cartridges for recovery tests, since this was a more exacting test of the technique.

2) "Florisil" Clean Up

The very large number of materials which, in real air samples may coeluete and interfere with the GC analysis, makes it absolutely necessary to perform a clean-up of the sample extract. In particular, the low chlorinated PCB isomers which are likely the most prevalent in the atmosphere due to their volatility are the isomers most interfered with. Recent results at MOE - ARB indicate that even the use of high resolution capillary gas chromatography may not resolve the numerous interfering materials and that a clean-up may simplify the interpretation and improve the accuracy of the resulting chromatogram.

The use of "Florisil" to clean up sample extracts is a common and accepted practice in both PCB and pesticide residue analysis. The clean-up was the last major hurdle and provided the most difficulties to completing the overall analytical scheme. The problem was compounded by the initial decision to use pentane rather than hexane throughout the program.

After having established a protocol for clean-up of the "Florisil", it was realized that the sample clean-up technique used by MOE -LSB (2) was not working as expected when the change to pentane was incorporated. The technique used by MOE - LSB involves utilizing a dry packed column of "Florisil". The "Florisil" was fully activated and allowed to pick up moisture (for deactivation) by sitting exposed to the atmosphere for a prescribed length of time (dependent on the relative humidity). The clean-up column was dry packed with this "deactivated Florisil", a sample carefully pipetted onto the top of the column, and additional pentane added until elution had progressed the length of the column. Twenty-five ml of pentane were then added to the top of the column and the elution allowed to proceed. Once we had assured ourselves that we had overcome the practical problems of handling volatile pentane which would vaporize and form cavities in the "Florisil" packed elution column, our results quickly indicated that quantitative recovery of some PCB isomers was not achieved. Isomers, which may be significant to the accuracy of the total PCB analysis, were being lost, e.g. 2-,4-,22',44' isomers.

The thermal oxidative cleanup of the "Florisil" produces a grade of "Florisil" which is more fully dehydrated and thus exhibits higher activity, than the material produced by continuous solvent extraction and activation at 135°C. Subsequent deactivation as practiced at MOE - LSB involving exposure of the "Florisil" to the atmosphere for a specific length of time depending on the measured humidity may not be applicable. A sample of thermally prepared "Florisil" (9.0 g) was exposed to the atmosphere (RH = 39%, T = 20°C) and weighed over a period of 30 minutes to monitor the amount of water absorbed. Under these conditions the weight gain was approximately 0.1% for every 5 min. and did not show signs of saturating after 30 min. (see fig. 6). It would take 2 1/2 hours to reach the level of 3% deactivation ultimately used in this study, during which time the "Florisil" may become contaminated as well. For these reasons it was decided to forego the procedure used at MOE - LSB; which in reality was designed to remove the bulk of PCB isomers as interferants in pesticides analysis; and specifically deactivate the "Florisil" with (a known quantity of) water, characterizing the clean-up to give quantitative recovery of PCB isomers.

A study of the elution profiles of PCB isomers on the clean-up column and the effect of deactivation of the "Florisil" with water was undertaken. (Refer to Appendix A for details on preparation of deactivated "Florisil"). Fully active "Florisil" prepared as described was deactivated with water. Three lots were prepared corresponding to one percent, two percent, and three percent of water by weight. After sufficient time was allowed for equilibration, a clean up column was dry packed for evaluation. An aliquot of mixture of 13 PCB isomers was placed on the column and the sample was further eluted with pentane. One millilitre fractions were collected in small vials for subsequent analysis by GC to determine the elution profiles of the isomers. Elution profiles determined are shown in Figures 7 to 10. Results are plotted as percentage of total recovered versus fraction number for each isomers tested. Fully activated "Florisil" significantly affected the elution of PCB isomers, some of which eluted very rapidly, e.g., (22'44'55'-), others within a larger volume, e.g., (44'-), and some were not recovered at all, e.g. (2-; 2,6-). The elution profile was drastically altered as the level of deactivation was changed from zero to one percent. Slightly evident changes were noticed in the elution profile as the level of deactivation was increased beyond one percent. Isomers which previously were not recovered, from fully active "Florisil", were detected and the elution volume became progressively shorter as deactivation increased. Even at 3 percent deactivation there is evidence that "Florisil" causes separation of PCB isomers. For fully active "Florisil", the general elution trend is such that the elution volume increases with decreasing number of chlorines. Steric hinderance of the two rings in the biphenyl system may also affect the retention; elution volume increasing with decreasing hinderance; although there are notable exceptions to this trend i.e. the lack of recovery of 2,6-dichlorobiphenyl. More work is indicated in this area.

A more detailed study was undertaken, during which the overall recovery of a larger number of isomers was characterized in both one and three percent deactivated "Florisil". Single batches of one percent and of three percent deactivated "Florisil" were prepared as described and stored in glass bottles with foil lined caps. This was sufficient material for the duration of the experiment. Each day for a period of a week three clean-up columns for each batch of "Forisil" were prepared. A 1 ml aliquot of a mixture of 25 PCB isomers used for instrumental calibration was spiked onto each of the elution columns. The isomers were eluted with pentane; three fractions of 10 ml each were collected from each of the columns. The volume of each fraction was reduced to 0.5 ml by evaporation using iso-octane as a keeper, and subsequently analyzed by GC using the same mixture of isomers for calibration of the GC. Overall recovery for one percent deactivated "Florisil" was low and variable; total recovery averaged 66% with a relative standard deviation of 18% for fourteen (14) runs. Using one percent deactivated "Florisil" various isomers (2-,4-,22'33'-) were constantly found in the second fraction (10 to 20 ml). Also some isomers such as 33'44-' were not recovered at all or on occasion trace amounts were present in the third fraction (20 to 30 ml). Results are shown in Table 10. Recovery for 3 percent deactivated "Florisil" was significantly higher and more reproducible, although some isomers such as 33'44-' do show variable recovery. The mean total recovery of all twenty-five (25) isomers tested was 99.8% with a relative standard deviation of 9% for twelve (12) recovery experiments. Results are given in Table 11. An elution volume of ten (10) ml was used in these experiments; a second ten (10) millilitre fraction showed only traces of PCB isomers which could not be quantitated. A brief experiment was conducted in which a series of elutions were done using 3% deactivated "Florisil" and collecting a total volume of 8, 10, 12, 14, 16 millilitres. Results of the recoveries of isomers in the 25 isomer mixture are given in Table 12, Problems were encountered with the analytical system during the course of this experiment which affected the results for the 14 ml and 16 ml elution volumes. A general trend can be seen (particularly for 33'44-23'5-, 22'66'-, 22'33'-) in which the recovery increases to a maximum for 12 ml. Elution volume of 14 ml was chosen as an optimum volume for sample clean-up permitting good recovery and allowing for any slight variation in "Florisil" activity during storage and/or preparation which may affect recovery. Results in Table 13 illustrate the repeatability of the clean-up recovery for 3% deactivated "Florisil" using a 14 ml pentane elution volume. Results for 2-monochlorobiphenyl are not presented in all cases due to problems with the digital integration and could not be estimated by peak height measurement due to the low sensitivity to 2-monochlorobiphenyl. Overall total recoveries (not corrected for the omission of 2-monochlorobiphenyl) average 79.6% with a relative standard deviation of 10.3% absolute. Our realization of the significance of the level of water deactivation on the elution profile of the PCB isomers prompted us to test the effect of water deactivation on the recovery of the PCB isomers from the field sampling cartridges. It was felt that initial tests to obtain quantitative recovery from sampling cartridges may have been affected by this factor. Six sets of three fully active cartridges were prepared and spiked with various PCB isomer mixtures (each member of a set being identically prepared). Two tubes in each set were then spiked with an aliquot of pure water equivalent to 1% and 3% of the weight of "Florisil" in the tube; sealed with the glass foot and sealing ring and stored so that the water would evaporate onto "Florisil". The PCB's were subsequently eluted from the cartridges using 200 ml of pentane; the extract concentrated in a "Kuderna-Danish" evaporator using 0.5 ml iso-octane keeper and analyzed by gas chromatography. Total results are given in Table 14 and do not indicated any consistent increase in recovery when the sampling cartridges are deactivated. Cartridges with high levels of water absorbed onto the "Florisil" are likely to simulate the actual state of the cartridges after sampling. The amount of water absorbed onto "Florisil" during sampling is potentially quite high. At a sampling temperature of 20°C and a relative humidity of 35% as much as 65 g of water (ten times the weight of "Florisil" present) will pass through the sampling cartridge when a 10 m^3 air sample is taken.

Recovery from actual field samples was not anticipated to give any problem due to the amount of water present. It was desired that in our program a series of quality control cartridges would be spiked with PCB's and utilized on a daily basis along with actual field survey samples. These cartridges would be fully activated unless these tests indicated that some deactivation of the "Florisil" was necessary.

The complete preparative and analytical methodology was subjected to (both) internal checks by the contractor and by the Ministry of Environment - Air Resources Branch. The results are summarized in Tables 15 and 16. These results were obtained by spiking of various PCB mixtures onto fully active sampling cartridges, elution with 200 ml of pentane, evaporation and clean-up on a (3% water deactivated) 100/200 mesh "Florisil" column and subsequent analysis by GC, using conditions described at the beginning of this report. The final results indicate that the overall clean-up and quantitation procedure does provide data which will be within 25 percent of the true value, 80 percent of the time.

A final check of the total assay was performed by analysis of cartridges which were spiked with PCB isomers and with a real air extract. A Hi-volume air sampler was set up outside of the contractors laboratory. Approximately 500 m^3 of air were sampled through a glass cartridge containing approximately 100 gms of precleaned "Florisil". This sample was extracted with 1 litre of pentane and concentrated to a final volume of 5 ml which was used for spiking on cartridges. A series of sixteen sampling cartridges were spiked with an aliquot of this Hi-Vol extract; fourteen of these were additionally spiked with mixtures of PCB isomers by MOE-ARB personnel. These cartridges were subsequently extracted and analyzed for total PCB's by the contractor.

Recoveries of the spiked PCB's ranged from 50% to 130% with an average figure of 69.2% for the 14 samples. Individual results are given in Table 17.

SELECTION OF STANDARDS

Previous studies in which stack emissions or ambient air have been monitored have indicated that the composition of PCB isomers in the sample are markedly different from that which is found in any of the commercial Arochlors (3,4). This is due, in part, to the wide range in volatility of the isomers that largely determines the vapor phase composition. Also, the large quantities of non PCB materials in the atmosphere which interfere, usually produce a chromatogram which bears no resemblance to any commercial Arochlor or combination of Arochlors.

Calibration methods utilizing commercial Arochlors for standards (5) but backed up by GC/MS for analyses would have been successful, but time consuming and prohibitively expensive considering the large number of samples to be encountered in this program.

Since the composition of Arochlors are not well defined and vary with their source and date of manufacture, it was decided that the calibration standard used for this program should be a cocktail which is a mixture of pure PCB isomers. This has distinct advantages:

i) The standard and its isomer composition are well defined, consequently calibration is more accurate
ii) The composition of the standard may be altered to reflect the source of PCB's in the atmosphere
iii) Can be altered if necessary to accommodate the analytical conditions used
iv) Permits a more detailed understanding of the problems associated with the analysis of PCB's.

The rules which are utilized for the selection of the isomers are given in Appendix A in Section F of the quality control program. These rules were formulated in an effort to meet the objectives mentioned.

Published data were used to generate the listing of major isomer which are found in commercial Arochlors (6 to 10). Utilizing this

listing and the rules, the only limitation to this method is that the isomer must be commercially available or easily synthesized, as well as reasonably pure.

For this last reason, 3-monochlorobiphenyl is not used in our standard mixture. The calibration mixture selected and being used is given in Table 18. This Table also gives the retention times under the conditions given earlier in this report.

SEALING OF AMPOULES

Ambient air samples which have been processed are sealed in glass ampoules for long term storage and transportation. Half of the sample extract in pentane is sealed for further analysis at the Ministry of Environment - Lab Services Branch. The remainder of the sample after undergoing a screening analyses for PCB's will be submitted to MOE-ARB in a sealed ampoule for further analysis.

The sealing procedure was tested in order to ensure that the ampoules could be sealed successfully. For small ampoules in which the final extract is sealed, an aliquot (0.5 ml) of an isomer mixture in isoctane was pipetted into an ampoule. The ampoule was chilled in a cryo-cooled bath of isopropanol and subsequently flame sealed. After storage for a day, the ampoules were reopened one at a time, analyzed by GC and the results directly compared against an analysis of the same isomer mixture which had been sealed in ampoules. At the higher concentration level (1123ng/ml) very good results were obtained (averaging 100.5% overall). The slightly lower results for the 4-mono isomer were due to a minor contamination in the vials which interfered with integration of this component peak. Similarly, reasonably good results were obtained at lower concentration levels (56 ng/ml) although these do show consistently high or low recoveries, being close to the detection limits for the isomer components. Some of the isomer peaks were not detected by the integrator system and are not reported. The larger ampoules (10 mls) in which pentane was used were somewhat more difficult to seal; these required thorough chilling before flame sealing. Speed was essential since the pentane quickly warmed up to exert a vapor pressure which would not permit the glass to fuse into a seal. It was not necessary to maintain the concentration of PCB isomers in the pentane. Thus overall recovery was of concern. An aliquot of (0.5 ml) a PCB isomer mixture was flame sealed into an ampoule along with 10 ml of pentane and stored for a day. The ampoule was then opened, quantitatively transferred to a micro "Snyder" concentrator, and reduced in volume to 0.5 ml. This concentrate was then analyzed by GC and compared directly with the mixture used for sealing. This sealing of pentane solutions in 10 ml ampoules which utilized considerably more heat then the 1.0 ml ampoules did not contribute to overall or selective loss of PCB's. The final results are given in Table 19.

Small vials which are used to contain samples prior to analysis on the GC were tested to determine the rate at which solvent vapour could pass through the seal in the lid. Five vials were filled with 0.5 ml of pentane folowed by sealing and weighing. Initial weight loss averaged 25.4 mg ± 23.6 mg/day of pentane which was unacceptably high and variable. Refridgeration was not as helpful as expected. Judicious tightening of the caps (more than one might normally apply), being careful to choose caps having liners which uniformly contact the rim of the vial reduced the weight loss to an average of 3.6 ± 2.4 mg/day of pentane from a vial containing 313 mg of pentane (0.5 ml). This was substantially less for iso-octane. A similar experiment over a 4 day period showed an average weight loss of iso-octane of 0.62 mg ± 0.43 mg/day over a four day period from vials containing 364 mg (0.5 ml) of iso-octane. These vials were considered to be suitable for short term storage while the samples are being analyzed. After analysis samples should be transferred to a glass ampoule and flame sealed.

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5) Quantitation of Polychlorinated Biphenyls Residues by Electron Capture Gas-Liquid Chromatography: Reference Material Characterization and Preliminary Study. Leon D. Sawyer J.A.O.A.C. V61 #2 pg. 272

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9) Comparison of the Compositions of Arochlor 1242 and Arochlor 1016
P. W. Albro, C. E. Parker, J. Chrom 169 (1979) pg.161

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DHEW (NIOSH) Publication No. 77-225

TABLE 1

Programme Listing For Varian CDS 111 Chromatography Data System

SECTION 1

1	ID#	37#
2	S/N	2N
3	IPW	9S
4	TAN%	20.00%
5	AREJ	500A
6	STOP	60.00M

SECTION 2

LINE	TIME	EUT	VALUE
1	.00	2FB	3.00
2	.00	8D1	10.00*
3	4.00	7PR	01
4	33.00	7PR	04
5	35.00	3W	28.00
6	38.00	3W	14.00
7	41.00	7PR	00
8	48.00	2FB	48.00
9	60.00	2FB	60.00

TABLE 2

Linearity and Precision Tests. The Concentration of Standard Mixture is Equivalent to 148.6 ng/m^3 of Total PCBs for X1 Area Counts. For the X100 Dilution, the symbol - indicates immeasurable signal.

Isomer	Peak No.	X1 Area Counts Mean	%SD	X10 Area Counts Mean	Ratio of Means 1/10	X100 Area Counts Mean	Ratio of Means 1/100
2	1	253614	15.5%	22375	11.33	-	-
4	2	235020	10.3%	20258	11.60	-	-
22'	3	296827	16.5	24596	12.07	-	-
24	4	535138	7.03	46960	11.40	-	-
23	5	1,108710	1.75	94092	11.78	5891	188
35	6	1,500632	4.09	117290	12.79	2633	570
22'5	7	1,185816	3.98	95012	12.48	6359	186
44'	8	1,094679	5.42	82456	13.28	-	-
22'66'	9	1,849287	0.86	155430	11.90	8109	228
23'5	10	799584	5.27	72568	11.02	-	-
22'56	11	2,046753	0.72	168337	12.16	-	-
23'4	12	3,128342	0.88	235824	13.27	3184	983
22'55'	13	1,317863	0.70	116206	11.34	-	-
22'44'	14	1,066899	2.24	95770	11.14	-	-
22'3'5	15	2,347715	0.94	190922	12.30	1115	2105
23'55'	16	5,020788	2.62	336518	14.92	16147	311
22'33'	17	4,374412	3.21	313356	13.96	18899	232
23'4'5	18	956165	0.53	92870	10.30	-	-
22'455	19	2,618761	1.99	218231	12.00	-	-
23'44'6	20	2,082612	0.95	171096	12.17	-	-
22'33'66'	21	5,126639	4.36	343198	14.94	9038	567
22'345'	22	3,909444	1.74	290866	13.44	6405	610
33'44'	23	3,112133	3.23	59072	52.68	-	-
22'44'55'	24	1,719122	4.03	37694	45.61	-	-
22'33'44'	25	1,165557	16.02	109442	10.65	7475	156

TABLE 3

Precision of Retention Times (RT) for Repeated Injections as Measured by the Mean, Standard Deviation (SD) and % Variation CV = (S.D./Mean) X100

Peak No.	RT Inj. 1	RT Inj. 2	RT Inj. 3	Mean	S. D.	C. V.
1	5.50	5.49	5.51	5.50	0.01	0.18
2	7.52	7.51	7.57	7.53	0.03	0.43
3	8.15	8.13	8.18	8.15	0.025	0.31
4	8.71	8.69	8.75	8.72	0.03	0.35
5	9.78	9.75	9.82	9.78	0.03	0.35
6 + 7	11.34	11.30	11.38	11.34	0.04	0.35
8	12.35	12.34	12.41	12.37	0.04	0.31
9	14.30	14.26	14.36	14.30	0.05	0.35
10	15.05	15.01	15.10	15.05	0.045	0.30
11	15.55	15.52	15.61	15.56	0.05	0.29
12	16.89	16.88	16.97	16.91	0.049	0.28
13	18.27	18.26	18.36	18.29	0.05	0.30
14	19.11	19.07	19.19	19.12	0.06	0.32
15	19.66	19.62	19.74	19.67	0.06	0.31
16	20.91	20.87	20.99	20.92	0.06	0.29
17	22.66	22.62	22.75	22.67	0.06	0.29
18	23.74	23.71	23.85	23.76	0.07	0.27
19	25.80	25.78	25.91	25.83	0.07	0.27
20	27.32	27.29	27.43	27.34	0.07	0.27
21	28.27	28.23	28.38	28.29	0.08	0.27
22	28.94	28.90	29.05	28.96	0.07	0.27
23	31.76	31.73	31.89	31.79	0.08	0.27
24	34.91	34.89	35.02	34.94	0.07	0.20
25	36.52	36.52	36.66	36.56	0.08	0.22
26	37.47	37.45	37.56	37.49	0.06	0.16
27 + 28	38.46	38.44	38.57	38.49	0.07	0.18
29	40.33	40.33	40.41	40.35	0.05	0.11
30	43.55	43.53	43.64	43.57	0.06	0.13
31	50.89	49.10	49.26	49.75	0.99	1.99

TABLE 4

Precision of Area Counts for Repeated Injections as Measured by the Mean, Standard Deviation (S.D.) and % Variation (C.V.) = (S.D./Mean) X100

Peak No.	Area Inj. 1	Area Inj. 2	Area Inj. 3	Mean	S.D.	C.V.
1	393914	485888	655140	511647	132504	25.9
2	163208	166952	184504	141555	11370	6.6
3	2126754	2145176	2513776	2595235	795532	30.7
4	86496	91540	96342	91459	4923	5.4
5	4722012	4767700	5562168	5017293	472428	9.4
6	5015264	4799542	5755754	5190186	501531	9.7
8	970984	1150080	1103452	1074839	92913	8.6
9	2984032	1855372	3288332	2976032	272825	9.2
10	1144164	1094252	1250128	1162848	79600	6.8
11	968820	966952	1085416	1007063	67862	6.7
12	1203552	1193592	1348608	1248584	86766	6.9
13	6061740	6177484	6892568	6377264	450003	7.1
14	3219686	3229106	3667642	3372144	255952	7.6
15	2471014	2519100	2768718	2586277	159817	6.2
16	3485792	3514284	3832460	3610845	192452	5.3
17	8265734	8319624	8791614	8458991	289318	3.4
18	16590652	16831456	17465172	16962427	451731	2.7
19	3954466	4015604	4115910	4028660	81510	2.0
20	14990740	15159918	16174562	15441740	640255	4.1
21	4269738	4350086	4843020	4487615	310401	6.9
22	3930336	3986758	4363378	4093491	235425	5.8
23	18774750	19045604	20868616	19562990	1138787	5.8
24	422578	5953648	6530536	5569988	1199321	21.5
25	11094792	10348656	11281432	10908293	473563	4.5
26	5176036	4938800	5390792	5168543	226089	4.4
27 + 28	18047476	16891056	18796244	17911592	959835	5.4
29	22088592	19997872	19927108	20671191	1228015	5.9
30	28056032	25556648	26407488	26673387	1270731	4.8
31	28180548	31508108	31471960	30386872	1910818	6.3

TABLE 5

Reproducibility of Retention Times
Using Varian 8000 Autosampler/Injector.
The parameters S.D. and C.V. are the average
standard deviation and the percent variation, respectively.

RT NO.	Injection Number							
	1	2	3	4	5	6	7	8
1	5.70	5.70	5.88	5.79	5.74	5.83	5.78	5.88
2	6.79	6.78	7.01	6.90	6.82	6.94	6.90	6.99
3	8.82	8.81	7.12	8.97	8.86	9.02	8.98	9.07
4	10.98	10.97	11.36	11.17	11.03	11.22	11.20	11.25
5	13.62	13.62	14.68	13.86	13.70	13.91	13.89	13.97
6	14.66	14.66	15.15	14.83	14.75	14.98	14.96	15.03
7	17.19	17.18	17.74	17.49	17.25	17.54	17.53	17.60
8	18.28	18.25	18.85	19.60	18.38	18.64	18.63	18.70
9	21.37	21.37	22.02	21.75	21.50	21.76	21.78	21.82
10	22.68	22.68	23.35	23.08	22.81	23.09	23.11	23.15
11	25.40	25.40	26.13	25.8	25.53	25.84	25.86	25.90
12	31.05	36.02	31.56	31.53	31.15	31.49	31.56	31.55
13	35.26	35.24	35.87	35.69	35.37	35.65	35.72	35.68

RT NO.	Mean (Min)	S.D. Min	C.V.
1	5.78	.062	1.07
2	6.89	.088	1.28
3	8.96	.116	1.29
4	11.15	.144	1.29
5	13.83	.168	1.22
6	14.89	.177	1.18
7	17.44	.204	1.17
8	18.66	.433	2.32
9	21.67	.233	1.08
10	22.99	.243	1.06
11	25.74	.263	1.02
12	31.40	.296	0.94
13	35.57	.256	0.72

TABLE 6

Precision of Peak Areas Using Varian 8000 Auto Sample/Injector.

(For explanation of symbols see Table 5).

RT NO.	DIL 1X	DIL 10X 1	DIL 10X 2	DIL 10X 3	DIL 10X 4	MEAN X	CV
1	204662	17854	18102	17567	17202	17631	2.37
2	6105282	202441	220768	204305	203226	207685	4.22
3	1012572	79045	89352	77661	77855	80978	6.94
4	980700	69458	80118	74102	69367	73261	6.93
5	4462918	259366	280508	264274	262418	266642	3.55
6	2702012	190762	209306	198972	194544	198396	4.04
7	20110200	499880	543306	510424	516628	517560	3.58
8	6145504	328584	353980	330767	330204	335861	3.61
9	22400836	533029	589388	548858	556172	556862	4.26
10	5486584	319192	348860	332372	320692	330279	4.15
11	10269292	405164	445108	420676	423062	421003	4.10
12	3786472	262216	287508	263996	263560	274320	4.76
13	15068634	319820	320164	339836	349040	332215	4.40

	DIL 5X 1	DIL 5X 2	DIL 5X 3	DIL 5X 4	MEAN X	CV
1	34431	34312	35719	35085	34887	1.86
2	411232	426605	420703	428163	421676	1.82
3	165124	167406	167120	165754	166351	0.66
4	150016	153900	154038	149332	151822	1.64
5	518046	537936	529776	533742	529875	1.62
6	385116	395672	392318	395042	385287	4.72
7	1014798	1065049	1044088	1053836	1044442	2.06
8	658208	685952	682298	687224	678421	2.01
9	1087728	1139209	1115300	1130684	1118230	2.02
10	643212	668044	649304	675152	658928	2.29
11	830184	885112	852452	878496	861561	2.93
12	531928	561120	547088	550060	547549	2.20
13	850128	842880	818760	809832	830400	2.31

TABLE 7

Detection limits for the various isomers on Varian 3700 using an electron capture detector

	PRACTICAL DETECTION LIMIT (pg)	ABSOLUTE DETECTION LIMIT (pg)
2	1.92	0.15
4	5.55	0.52
22'	1.60	0.14
24	0.12	0.011
23	0.12	0.015
35	0.30	0.043
22'5	0.19	0.028
44'	1.01	0.20
22'66'	0.24	0.041
23'5	0.11	0.016
22'56	0.14	0.026
23'4	0.088	0.018
22'55'	0.146	0.027
22'44'	0.082	0.016
22'3'5	0.080	0.017
23'55'	0.038	0.011
22'33'	0.054	0.008
23'4'5	0.073	0.016
22'455'	0.061	0.014
23'44'6	0.043	0.011
22'33'66'	0.069	0.025
22'345'	0.037	0.012
33'44'	0.061	0.017
22'44'55'	0.029	0.003
22'33'44'	0.042	0.011
Total	12.20	1.39

TABLE 8

The Analytical System Stability

	Retention Time		Calibration Factor	
	Mean	% S. D.	Mean	% S. D.
2	3.61	0.52 %	6.280	6.2 %
4	5.03	0.45	16.395	5.4
22'	5.53	0.47	6.001	11.1
24	6.68	0.38	0.359	6.0
23	7.76	0.40	0.383	6.6
35	8.54	0.28	0.982	6.6
22'5	9.96	0.34	0.646	7.1
44'	11.00	0.27	3.077	4.1
22'66'	11.97	0.32	0.825	8.6
23'5	13.21	0.25	0.327	5.8
22'56	14.22	0.29	0.462	8.1
23'4'	15.29	0.23	0.282	6.6
22'55'	16.90	0.27	0.463	7.1
22'44'	17.74	0.22	0.258	7.1
22'3'5	19.37	0.20	0.257	7.4
22'55'	20.67	0.21	0.127	6.5
22'33'	22.04	0.21	0.194	8.6
23'4'5	24.73	0.17	0.191	5.4
22'455'	27.49	0.17	0.184	6.4
23'44'6	29.21	0.17	0.130	6.2
22'33'66'	30.28	0.17	0.259	9.2
22'345'	31.24	0.17	0.124	7.7
33'44'	34.52	0.12	0.186	6.8
22'44'55'	38.71	0.09	0.096	5.1
22'33'44'	43.23	0.09	0.107	6.7

TABLE 9

Recovery of PCB Isomers Using "Kuderna-Danish" Evaporators

% Recovery

Peak No.	RT	STD 1	STD 2	STD 3
1	6.75	95		99
2	9.53			100
3	10.35		124	181*
4	10.75			102
5	12.04			101
6	13.51	98		
7	14.01	95		
8	14.88	94		100
9	17.90	93		99
10	18.37		98	
11	19.66		110	100
12	20.67			103
13	21.95	92		101
14	23.18	92	98	125
15	24.18			
16	24.83	91	98	100
17	25.64			101
18	27.23	94		100
19	27.87	94	98	
	28.39		102	
20	30.78		98	
21	31.77		96	
22	33.83	95		101
23	34.55		96	
24	36.02	94	97	
25	37.87	94		103
26	38.72			101
27	40.06	88		101
28	40.97		105	
29	43.15		82	
30	44.67		97	
	46.10		103	
	48.99		103	102
31	51.06		100	102
TOTALS	mean	93.5	100.1	106.1
	std. dev.	2.1	8.59	18.47
	rel. std. dev.	2.47%	8.58%	17.41%

* Analysis of this component is high due to a coeluting component. Actual peak size is small relative to other components.

TABLE 10

Recovery of Spiked PCB Isomers From 1% Water Deactivated "Florisil" Clean-up Column, the indices 1 and 2 refer to the first and second 10 ml fractions of eluant.

Isomer		Amount	A-1	A-2	%	B-1	B-2	%	C-1	C-2	%
2	1	9.72		6.64	(68)		7.17	(74)		6.85	(70)
4	2	26.1	8.65	6.11	(56)		18.28	(70)			(0)
22'	3	9.51		5.82	(61)		9.01	(95)		5.15	(54)
24	4	1.30	0.95		(73)	1.30		(100)	1.10		(85)
23	5	2.73		2.04	(75)		2.19	(80)		2.20	(81)
35	6	9.03	7.17		(79)	7.17		(85)	7.00		(78)
22'5	7	4.55	3.92		(86)	4.72		(104)	4.35		(96)
44'	8	24.03	13.1		(55)	8.32	6.66	(62)			(0)
22'66'	9	8.80	8.20		(93)	7.99		(91)	10.10		(115)
23'5	10	1.72	1.35		(78)	1.69		(98)	1.40		(81)
22'56'	11	5.63	5.50		(98)	6.16		(109)	6.10		(108)
23'4	12	5.48	4.40		(80)	5.13		(94)	4.25		(78)
22'55'	13	3.85	3.23		(84)	3.68		(96)	3.70		(96)
22'44'	14	1.77	1.36		(77)	1.72		(97)	1.40		(79)
22'3'5	15	3.76	3.18		(85)	3.53		(94)	3.45		(92)
23'55'	16	3.83	3.02		(79)	3.27		(85)	3.50		(91)
22'33'	17	4.76		3.26	(68)		2.99	(63)		3.20	(67)
23'4'5	18	1.40	0.95		(68)	1.33		(95)	0.75		(54)
22'455'	19	3.18	2.70		(85)	3.02		(95)	3.25		(102)
23'44'6	20	1.80	0.89		(49)	1.72		(96)	1.50		(83)
22'33'66'	21	7.10	5.93		(84)	6.38		(90)	7.40		(104)
22'345'	22	2.92	1.96		(67)	2.72		(93)	1.75		(60)
33'44'	23	3.80		2.10	(55)		1.14	(30)		3.02	(79)
22'41'55'	24	1.00	0.66		(66)	0.79		(79)	0.88		(88)
22'33'44'	25	0.98	-	-	(0)		0.58	(59)	0.49		(50)
TOTAL		148.7	77.1	26.0	(69)	71.1	48.00	(81)	62.4	20.4	(56)

TABLE 10 (Cont'd)

D-1	D-2	%	E-1	E-2	%	F-1	F-2	%	G-1	G-2	%
4.50	2.20	(69)		7.50	(77)	2.95	21.94	(128)	4.57	20.20	(127)
		(0)			(0)	0	0	(0)		0	(0)
8.95	2.20	(117)		4.46	(47)		6.46	(68)		5.30	(56)
0.85		(65)	0.88		(69)						
0.85	1.20	(75)		1.82	(67)		2.48	(91)		2.00	(73)
6.15		(68)	6.51		(72)						
3.75		(82)	3.91		(86)						
19.31		(80)	10.81		(45)	12.02		(52)	13.22		(57)
9.65		(110)	10.53		(120)	11.08		(126)	12.17		(142)
1.20		(70)	0.89		(52)						
5.15		(91)	5.14		(97)						
3.75		(68)	4.04		(74)						
2.80		(73)	3.19		(83)						
1.05		(59)	1.20		(68)						
2.85		(76)	3.59		(95)						
2.6		(68)	3.42		(89)						
	2.95	(63)		3.63	(76)		4.63	(97)		4.40	(92)
0.60		(93)	0.64		(46)						
2.75		(86)	3.28		(103)						
1.50		(83)	1.54		(86)						
5.45		(77)	7.43		(105)						
1.65		(57)	1.81		(63)						
	4.32	(114)			(0)		2.80	(74)		3.02	(79)
0.60		(60)	4.84		(484)						
0.60		(61)	5.12		(522)						
67.25	12.87	(54)	68.77	1 7.41	(58)	26.05	38.31	(83)	30.26	34.92	(84)

TABLE 10 (Cont'd)

	J-1	J-2	%	K-1	K-2	%	M-1	M-2	%
1		13.88	(137)		8.67	(89)		6.83	(70)
2		10.87	(42)		19.57	(75)		0	(0)
3		6.54	(69)		5.67	(60)		5.67	(60)
4	1.02		(78)	0.75		(58)	0.96		(74)
5		2.33	(85)		2.37	(87)		2.21	(81)
6	7.15		(79)	6.52		(72)	7.24		(80)
7	4.20		(92)	3.57		(78)	4.20		(92)
8		0	(0)		18.20	(76)		18.20	(76)
9	6.25		(71)	3.61	7.18	(123)	11.36		(129)
10	1.04		(60)	0.91		(53)	1.05		(61)
11	5.25		(93)	5.06		(90)	5.18		(93)
12	4.19		(76)	3.18	1.11	(78)	4.62		(84)
13	3.26		(85)	3.07		(80)	3.22		(88)
14	1.25		(71)	1.22		(70)	1.33		(75)
15	3.18		(85)	2.44	0.79	(86)	3.56		(95)
16	313		(82)	2.97		(78)	3.24		(85)
17		3.67	(77)		3.87	(81)		3.52	(74)
18		0	(0)	0.80		(57)	0.26		(19)
19	2.85		(90)	2.70		(85)	2.92	(92)	
20	1.27		(71)	1.23		(68)	1.36		(76)
21	6.63		(93)	6.66		(94)	6.82		(96)
22	1.82		(62)	1.58		(54)	1.87		(64)
23		2.07	(54)		1.58	(42)		4.27	(112)
24	0.69		(69)	0.68		(68)	0.74		(74)
25		0	0		0	(0)	0.49		(50)
TOTAL	53.18	38.76	(62)	46.96	69.69	(78)	60.52	41.05	(68)

TABLE 10 (Cont'd)

N-1	N-2	%	O-1	O-2	%	P-1	P-2	%	R-1	R-2	%
	10.20	(105)		7.76	(80)		7.66	(79)			
		(0)		0	(0)		15.22	(58)	13.32		(51)
1.28		(98)	0.92		(71)	0.97		(75)	1.35		(104)
	2.27	(83)		2.37	(87)		2.19	(80)		2.61	(96)
7.90		(87)	6.61		(73)	7.22		(80)	8.24		(91)
4.52		(99)	3.80		(66)		4.48		(98)	4.95	(109)
11.15		(46)		0	(0)	9.39		(39)	15.92		(66)
7.31		(83)	9.85		(112)	5.78		(66)	8.73		(99)
1.63		(95)	0.98		(57)	1.05		(61)	1.76		(102)
5.72		(102)	4.64		(82)	5.47		(97)	6.27		(111)
5.03		(92)	4.00		(73)	4.83		(88)	5.36		(98)
3.93		(102)	2.94		(76)	3.61		(94)	3.90		(101)
1.79		(101)	1.11		(63)	1.42		(80)	1.78		(101)
3.53		(94)	2.97		(79)	3.79		(101)	3.71		(99)
3.30		(86)	2.75		(72)	3.63		(95)	3.73		(97)
	3.52	(74)		4.16	(87)		3.55	(75)		3.70	(78)
1.37		(98)		0	(0)	0.81		(58)	1.49		(106)
2.98		(94)	2.50		(79)	3.22		(101)	3.17		(100)
1.73		(96)		0	(0)		0	(0)	1.81		(101)
6.28		(88)	4.90		(69)	6.36		(90)	6.71		(95)
2.63		(90)	1.40		(48)	1.97		(67)	2.80		(99)
	2.11	(56)	0		(0)		0	(0)		2.31	(61)
0.74		(74)	0.61		(61)	0.76		(76)	0.69		(69)
0.45	0.32	(79)		0	(0)		0.80	(82)	0.43	0.38	(83)
73.23	27.18	(68)	49.98	20.07	(47)	64.76	35.60	(67)	96.06	26.82	(83)

TABLE 10 (Cont'd)

	W-1	W-2	%	Y-1	Y-2	%	Mean Recovery		% S_D
							ng.	%	
1		5.93	(61)	5.57		(57)	7.72	(79)	25.45
2			0	27.12		(104)	8.51	(33)	112.1
3		8.32	(87)	6.98		(73)	7.21	(76)	27.3
4	1.23		(95)	1.35		(104)	1.07	(82)	18.9
5		2.30	(84)	1.18		(43)	2.15	(79)	15.6
6	7.28		(81)	8.38		(93)	7.22	(80)	9.1
7	4.27		(94)	4.53		(100)	4.23	(93)	9.4
8			-	14.85		(62)	10.42	(43)	7111
9	6.60		(75)	7.50		(85)	8.62	(98)	20.9
10	1.12		(65)	1.75		(102)	1.27	(74)	25.2
11	4.30		(76)	5.48		(97)	5.39	(96)	10.4
12	4.54		(83)	5.09		(93)	4.54	(83)	10.8
13	3.59		(93)	3.84		(100)	3.43	(105)	10.6
14	1.67		(94)	1.81		(102)	1.44	(81)	18.6
15	3.40		(90)	3.62		(96)	3.40	(107)	8.2
16	3.18		(83)	3.33		(87)	3.33	(84)	9.7
17		3.44	(72)		1.22	(26)	3.35	(70)	20.8
18	1.41		(101)	1.46		(104)	0.85	(61)	61.4
19	2.85		(90)	3.17		(100)	2.95	(93)	8.2
20	1.68		(93)	2.93		(100)	2.11	(72)	24.7
21	6.13		(86)	6.59		(93)	6.41	(90)	10.6
22	2.71		(93)	2.93		(100)	2.11	(72)	24.7
23		2.39	(63)		1.50	(39)	1.92	(50)	72.3
24	0.88		(88)	0.91		(91)	0.74	(74)	13.6
25			-	0.92		(94)	0.37	(37)	90.2
TOTAL	56.80	22.37	(53)	120.10	2.72	(83)	98.4	(66)	17.7

TABLE 11

Recovery of Spiked PCB Isomers From 3% Water Deactivated "Florisil" Clean-up Column, One Elution Volume of 10ml used in these experiment. A second Fraction, of 10ml showed no quantifiable PCB traces.

Isomer		Spiked Amt	AA-1		BB-1		CC-1		DD-1		EE-1		FF-1		GG-1		HH-1	
		ng	ng	%	ng	%	ng	%	ng	%	ng	%	ng	%	ng	%	ng	%
2	1	9.72	5.69	(58)	17.74	(183)	8.12	(83)	1.01	(10)	8.85	(91)	15.44	(159)	11.29	(116)	9.99	(103)
4	2	26.09	27.08	(104)	27.60	(102)	27.77	(106)	27.15	(104)	27.09	(104)	34.14	(131)	33.09	(127)	31.85	(122)
22'	3	9.51	8.94	(94)	9.37	(99)	9.46	(99)	9.17	(96)	9.06	(95)	11.33	(119)	11.41	(120)	10.71	(113)
24	4	1.30	1.16	(89)	1.25	(96)	1.25	(96)	1.28	(98)	1.23	(94)	1.48	(114)	1.50	(115)	1.42	(109)
23	5	2.73	1.89	(67)	2.31	(84)	2.37	(87)	2.49	(90)	2.38	(87)	2.75	(101)	2.82	(103)	2.68	(98)
35	6	9.03	6.61	(73)	8.31	(92)	8.29	(92)	8.42	(93)	8.38	(93)	9.82	(109)	9.92	(110)	8.45	(94)
22'5	7	4.55	4.21	(92)	4.44	(98)	4.53	(100)	4.66	(102)	4.65	(102)	5.17	(114)	5.01	(110)	4.63	(102)
44'	8	24.03	15.18	(63)	23.21	(97)	23.20	(97)	23.23	(97)	24.29	(101)	25.89	(108)	19.57	(81)	17.79	(74)
22'66'	9	8.80	6.22	(71)	8.60	(98)	8.71	(99)	8.90	(101)	8.93	(101)	10.04	(114)	8.67	(98)	7.46	(85)
23'5	10	1.72	1.24	(72)	1.58	(92)	1.64	(95)	1.68	(98)	1.65	(96)	1.79	(104)	1.87	(109)	1.70	(99)
22'56'	11	5.63	5.53	(98)	5.55	(98)	5.65	(100)	6.16	(109)	6.36	(113)	6.77	(120)	6.50	(115)	5.80	(103)
23'4'	12	5.48	4.49	(82)	4.76	(87)	4.86	(89)	5.27	(96)	5.22	(95)	5.59	(102)	5.63	(103)	5.22	(95)
22'55'	13	3.85	3.60	(94)	3.59	(93)	3.62	(94)	4.03	(105)	3.91	(102)	4.05	(105)	4.14	(107)	4.00	(104)
22'44'	14	1.77	1.68	(95)	1.70	(96)	1.72	(97)	1.90	(107)	1.83	(103)	1.86	(105)	1.93	(109)	1.86	(105)
22'3'5	15	3.76	3.44	(91)	3.55	(94)	3.49	(93)	3.71	(99)	3.60	(96)	3.96	(105)	4.07	(108)	3.71	(99)
23'55'	16	3.83	3.18	(83)	3.41	(89)	3.30	(86)	3.39	(89)	3.36	(88)	3.72	(97)	3.75	(98)	3.42	(89)
22'33'	17	4.76	3.96	(83)	4.41	(93)	4.22	(89)	4.16	(87)	4.13	(87)	4.59	(96)	4.41	(93)	3.79	(80)
23'4'5'	18	1.40	1.46	(104)	1.40	(100)	1.55	(111)	1.45	(104)	1.39	(99)	1.55	(110)	1.47	(105)	1.28	(91)
22'4'55'	19	3.18	2.97	(93)	3.02	(95)	2.96	(93)	3.03	(95)	3.06	(96)	3.32	(104)	3.35	(105)	3.30	(95)
23'44'6	20	1.80	1.56	(87)	1.71	(95)	1.64	(91)	1.74	(96)	1.77	(98)	1.88	(104)	1.91	(106)	1.70	(94)
22'33'66'	21	7.10	5.81	(82)	6.20	(87)	6.16	(87)	6.38	(90)	6.36	(90)	6.97	(98)	6.97	(98)	6.34	(89)
22'345'	22	2.92	2.56	(88)	2.68	(92)	2.59	(89)	2.73	(93)	2.73	(93	2.97	(102)	3.05	(104)	2.71	(93)
33'44'	23	3.80	2.51	(66)	2.85	(75)	2.62	(69)	1.91	(50)	2.41	(63)	2.29	(60)	2.21	(58)	1.65	(43)
22'44'55'	24	1.00	0.82	(82)	0.86	(86)	0.74	(74)	0.76	(76)	0.71	(71)	0.81	(81)	0.80	(81)	0.73	(73)
22'33'44'	25	0.98	0.88	(90)	0.95	(97)	0.83	(85)	0.79	(81)	0.77	(79)	0.87	(89)	0.84	(85)	0.75	(77
TOTAL		148.7	122.6	(82)	151.0	(102)	141.3	(95)	135.3	(91)	288.2	(97)	337.7	(114)	312.2	(105)	285.00	(96)

TABLE 11 (Cont'd)

	II-1		JJ-1		KK-1		LL-1		Mean Recovery		
	ng	%	ng	%	ng	%	ng	%	ng	%	Rel. std. der.
1	3.12	(32)	12.39	(127)	10.41	(107)	11.80	(121)	9.65	(99)	49.%
2	29.30	(112)	32.74	(125)	32.08	(123)	33.07	(127)	30.25	(116)	9.3%
3	10.04	(106)	11.29	(119)	11.20	(118)	11.14	(117)	10.26	(108)	9.9%
4	1.33	(102)	1.44	(111)	1.47	(113)	1.44	(111)	1.35	(104)	8.6%
5	2.51	(92)	2.64	(97)	2.88	(105)	2.64	(97)	2.53	(93)	11.2%
6	8.54	(95)	9.23	(102)	8.84	(98)	9.08	(101)	8.66	(96)	9.9%
7	4.76	(105)	5.09	(112)	5.14	(113)	4.51	(99)	4.73	(104)	6.5%
8	23.92	(100)	26.03	(108)	27.17	(113)	22.34	(93)	22.65	(94)	15.6
9	9.25	(105)	10.07	(114)	10.53	(120)	9.36	(106)	8.90	(101)	13.2%
0	1.80	(104)	2.06	(119)	2.27	(132)	1.85	(108)	1.76	(102)	14.4%
1	5.79	(103)	6.28	(111)	6.55	(116)	5.91	(105)	6.07	(108)	6.9%
2	5.07	(93)	5.57	(102)	5.91	(108)	5.12	(93)	5.23	(95)	7.8%
3	3.75	(97)	4.20	(109)	4.57	(119)	3.83	(99)	3.94	(102)	7.3%
4	1.77	(100)	1.94	(109)	2.13	(120)	1.80	(102)	1.84	(104)	6.8%
5	3.69	(98)	3.83	(102)	4.15	(110)	3.77	(100)	3.75	(100)	5.9%
6	3.37	(89)	3.51	(92)	3.67	(96)	3.44	(90)	3.46	(90)	5.0%
7	3.97	(83)	4.27	(90)	4.44	(93)	3.66	(77)	4.17	(88)	6.7%
8	1.22	(87)	1.30	(93)	1.60	(114)	1.22	(87)	1.41	(101)	9.2%
9	3.13	(98)	3.07	(96)	3.21	(101)	3.04	(96)	3.10	(98)	4.2%
0	1.77	(98)	1.74	(96)	1.84	(102)	1.70	(94)	1.75	(97)	5.6%
1	6.44	(91)	6.49	(91)	6.69	(94)	6.34	(89)	6.43	(91)	5.1%
2	2.76	(95)	2.73	(93)	2.92	(100)	2.68	(92)	2.76	(95)	5.4%
3	1.99	(52)	2.15	(57)	1.84	(48)	1.22	(32)	2.14	(56)	21.1%
4	0.80	(80)	0.78	(78)	0.85	(85)	0.80	(80)	0.79	(78)	5.9%
4	0.93	(94)	0.91	(92)	0.87	(89)	0.89	(90)	0.86	(87)	7.3%
TAL	281.9	(95)	323.32	(109)	326.36	(110)	305.24	(103)	148.35	(99.8)	±8.84%

TABLE 12

Recovery of PCB Isomers As a Function of Elution Volume.

Isomer		Spiked ng	8mls ng	8mls %	10mls ng	10mls %	12mls ng	12mls %	14mls ng	14mls %	16mls ng	16mls %
2	1	9.72	-	-	7.52	(77)	9.42	(97)	4.89	(50)	4.67	(48)
4	2	26.09	27.21	(104)	30.97	(119)	31.92	(120)	24.45	(94)	21.40	(82)
22'	3	9.51	8.47	(89)	10.45	(110)	10.48	(110)	8.46	(90)	7.28	(77)
24	4	1.30	1.36	(105)	1.37	(105)	1.34	(103)	1.12	(86)	1.04	(80)
23	5	2.73	2.16	(79)	2.49	(91)	2.58	(95)	2.22	(81)	2.05	(75)
35	6	9.03	8.63	(96)	8.82	(97)	9.23	(102)	7.76	(86)	7.36	(81)
22'5	7	4.55	4.72	(104)	4.75	(104)	4.72	(104)	4.03	(89)	3.83	(84)
44'	8	24.03	17.02	(71)	14.39	(60)	17.43	(73)	13.88	(58)	11.55	(48)
22'66'	9	8.80	0.52	(6)	7.52	(85)	3.30	(37)			8.55	(97)
23'5	10	1.72	0.23	(13)	1.14	(66)	0.89	(51)	0.53	(31)		
22'56'	11	5.63	4.19	(74)	7.35	(130)	5.47	(97)	4.98	(88)	3.31	(59)
23'4'	12	5.48	5.04	(92)	5.80	(106)	5.38	(98)	4.61	(84)	3.84	(70)
22'55'	13	3.85	4.06	(105)	3.93	(102)	4.02	(104)	3.56	(92)	3.57	(93)
22'44'	14	1.77	1.96	(111)	1.84	(104)	1.92	(108)	1.69	(95)	1.83	(103)
22'3'5	15	3.76	4.27	(113)	4.01	(107)	4.27	(114)	3.82	(101)	4.21	(112)
23'55'	16	3.83	3.70	(97)	3.87	(101)	4.16	(108)	3.79	(99)	4.07	(106)
22'33'	17	4.76	0.18	(4)	3.65	(77)	5.60	(118)	4.59	(96)	5.09	107)
23'4'5	18	1.40	3.03	(216)	1.34	(95)	1.66	(118)	2.03	(145)	3.69	(260)
22'455'	19	3.18	5.87	(185)	3.38	(106)	3.93	(123)	3.78	(119)	5.45	(171)
23'44'6	20	1.80	2.84	(158)	1.92	(106)	2.03	(113)	1.93	(107)	2.54	(141)
22'33'66'	21	7.10	9.37	(132)	7.37	(104)	7.68	(108)	7.15	(101)	8.21	(116)
22'345'	22	2.92	5.34	(183)	3.20	(109)	3.18	(109)	3.33	(114)	4.06	(139)
33'44'	23	3.80			1.12	(29)	2.73	(72)	2.86	(75)	3.03	(80)
22'44'55'	24	1.00	0.92	(92)	0.86	(86)	0.93	(93)	0.89	(89)	0.94	(94)
22'33'44'	25	0.98	0.80	(82)	0.93	(94)	1.00	(102)	0.86	(88)	0.91	(93)
TOTAL		148.65	121.85	(82)	139.92	(94)	144.8	(97)	116.1	(78)	122.4	(82)

TABLE 13

Reproducibility of Clean-up Procedures As Tested by Recovery of Spiked PCB Isomers.

Isomer		Amt Spiked	FC-7		FC-8		FC-6		FC-9		FC-10	
			ng	%	ng	%	ng	%	ng	%	ng	%
2	1	9.72	0.46	(47)			3.13	(32)				
4	2	26.09	18.40	(71)	19.94	(76)	26.27	(101)	24.34	(93)	26.56	(102)
22'	3	9.51	7.32	(77)	6.66	(70)	11.12	(117)	9.55	(100)	10.12	(106)
24	4	1.30	0.91	(70)	0.99	(76)	1.16	(89)	1.03	(79)	1.26	(97)
23	5	2.73	2.13	(78)	2.24	(82)	2.78	(102)	2.29	(84)	2.70	(99)
35	6	9.03	6.58	(73)	6.56	(73)	8.29	(92)	7.24	(80)	8.93	(99)
22'5	7	4.55	3.91	(86)	3.49	(77)	4.30	(94)	4.07	(89)	4.81	(106)
44'	8	24.03	17.50	(73)	17.83	(74)	20.86	(87)	18.44	(77)	20.94	(87)
22'66	9	8.80	7.21	(82)	6.96	(79)	8.48	(96)	9.30	(106)	9.23	(105)
23'5	10	1.72	0.90	(52)	1.29	(75)	1.53	(88)	0.95	(55)	1.50	(87)
22'56'	11	5.63	4.61	(82)	4.31	(76)	5.23	(93)	4.86	(86)	5.38	(96)
23'4	12	5.98	4.09	(75)	4.23	(77)	5.22	(95)	4.72	(86)	5.28	(96)
22'55'	13	3.85	2.97	(77)	3.07	(80)	3.70	(96)	3.48	(90)	3.79	(98)
22'44'	14	1.77	1.41	(80)	1.41	(80)	1.75	(99)	1.67	(94)	1.78	(100)
22'3'5'	15	3.76	2.94	(78)	2.80	(74)	3.55	(94)	3.35	(89)	3.66	(97)
23'55'	16	3.83	2.84	(74)	2.77	(72)	3.43	(90)	3.18	(83)	3.53	(92)
22'33'	17	4.76	3.34	(70)	3.29	(69)	4.15	(87)	3.92	(82)	4.27	(90)
23'4'5'	18	1.40	1.12	(80)	0.87	(62)	1.40	(98)	1.13	(81)	1.37	(98)
22'455'	19	3.18	2.25	(71)	2.13	(67)	2.62	(82)	2.48	(78)	2.72	(85)
23'44'6'	20	1.80	1.38	(77)	1.33	(74)	1.60	(87)	1.56	(87)	1.64	(91)
22'33'66'	21	7.10	5.42	(76)	5.19	(73)	6.39	(90)	6.15	(87)	6.62	(93)
22'345'	22	2.92	2.25	(77)	2.14	(73)	2.63	(90)	2.49	(85)	2.72	(93)
33'44'	23	3.80	1.72	(45)	1.92	(51)	2.10	(55)	1.99	(52)	2.14	(56)
22'44'55'	24	1.00	0.52	(52)	0.44	(44)	0.54	(54)	0.98	(98)	1.01	(101)
22'33'44'	25	0.98	0.87	(88)	0.76	(78)	0.97	(99)	0.85	(87)	0.89	(91)
TOTAL		148.65	102.96	(69)	133.12	(90)	119.98	(81)	132.77	(89)	132.85	(90)

TABLE 14

Recovery of PCBs from Water Deactivated Sampling Cartridges.

Cartridge Series No.	Amount Spiked ng	Amount Recovered (ng)		
		0% Deactivated	1% Deactivated	3% Deactivated
30	890	934	900	1118
40	940	606	635	649
50	746		Samples Lost	
60	1382	930	836	805
70	744	835	745	831
80	704	679	759	717

TABLE 15

Recovery Internal Quality Control Checks

Experiment No.	Quantity Spiked (ng)	Quantity Recovered (ng)	Percentage Recovery
IC-1	90.0	71.7	80
IC-2	111.2	87.0	78
IC-3	46.2	36.1	78
IC-4	44.1	37.5	85
IC-5	35.3	29.7	84
IC-6	Blank	0.0	-
IC-7	60.0	67.3	112
IC-8	43.2	37.8	87
IC-9	48.1	42.2	88
IC-10	122.2	112.2	92
IC-11	77.7	70.2	90
IC-12	71.9	78.4	109

TABLE 16

Results of MOE Quality Control Cartridges

		GC-3			GC-4			GC-5	
	Spiked ng	recovered ng	Mean %	Spiked ng	recovered ng	Mean %	spiked ng	recovered ng	Mean %
1							38.9	37.8	(97)
2	34.8	31.3	(90)						
3	38.0	38.7	(102)						
4	53.4	50.1	(94)						
5									
6	36.1	37.7	(104)						
7				36.4	31.3	(86)			
8							38.4	56.9	(148)
9									
10				34.3	74.1	(216)			
11				37.5	52.1	(139)			
12							36.5	47.2	(129)
13									
14							35.3	51.4	
15							37.6	47.7	(127)
16									
17									
18	37.2	31.0	(83)						
19	31.8	26.0	(82)						
20	36.0	34.8	(97)						
21	35.5	33.4	(94)	35.5	40.5	(114)			
22									
23	38.0	32.4	(85)						
24							39.8	47.7	(120)
25									
TOTAL	340.8	315.4	(93)	143.7	198.0	(137)	226.5	288.7	(127)

TABLE 17

Total Recoveries of PCBs From Cartridges Spiked with Hi-Vol Air Extract

Sample #	Total Spiked (ng)	Recovery (ng)	%
3A	445.3	225.7	50.7
3B		226.7	51.0
4A	470.6	339.0	72.0
4B		239.4	50.9
5A	373.2	485.7	130.2
5B		420.6	113.0
6A	696.0	348.8	50.1
6B		382.5	55.0
7A	372.1	330.8	88.9
7B		251.3	67.5
8A	352.3	271.8	77.2
8B		215.5	61.1
9A	380.8	189.5	49.8
9B		213.1	55.9

Average of % Recovery = 69.5% ± 25.3%

TABLE 18
Composition of Stock Calibration Mixture

NO	ISOMER	RETENTION TIME	CONCENTRATION
1	2	4.43 min	19.43 ug/ml
2	4	6.21	26.10
3	22'	6.79	9.51
4	24	8.18	2.67
5	23	9.47	2.73
6	35	10.40	9.03
7	22'5	12.04	5.45
8	44'	13.31	24.03
9	22'66'	14.35	8.80
10	23'5	15.80	3.43
11	22'56'	16.94	5.63
12	23'4'	18.17	3.65
13	22'55'	19.95	5.79
14	22'44'	20.86	3.53
15	22'3'5	22.67	3.29
16	23'55'	24.11	1.92
17	22'44'6	25.63	1.49
18	23'4'5	28.54	3.72
19	22'455'	31.53	3.18
20	23'44'6	33.37	1.80
21	22'33'66'	34.43	3.55
22	22'345'	35.34	2.92
23	33'44'	38.00	3.80
24	22'44'55'	41.27	1.00
25	22'33'44'	46.07	0.98
		TOTAL	159.1 ug/ml

TABLE 19:

Results of the Vial Sealing Experiments

		Iso-octane 1.0 ml vial 1124 ng/ml				Iso-octane 1.0 ml vial 56 ng/ml				Pentane 10 ml vial 491 ng/ml		
		V1	V2	V3	V4	V1	V2	V3	V4	V1	V2	V3
	1	88.4	88.5	88.3	89.8	102.1	123.4	104.9	134.5	114.4	108.3	108.2
	2	103.6	105.0	103.5	102.8	102.1	124.8	114.0	139.4	102.8	97.6	105.6
	3	103.9	91.1	102.4	91.5	104.0	126.4	110.7	143.5	102.7	87.9	105.4
5	4	98.1	99.0	103.6	97.7	97.9	116.8	102.9	139.1	104.2	96.3	96.9
66'	5	100.9	97.9	93.6	93.0	96.6	125.1	99.0	152.8	111.3	103.9	109.2
'	6	99.9	96.7	99.7	97.4	90.1	119.8	97.7	132.4	103.6	96.4	102.6
55'	7	102.6	106.1	104.5	101.9	91.1	124.2	101.0	129.4	102.6	96.9	102.1
3'5	8	102.3	105.9	109.8	102.4	94.3	122.2	103.1	137.9	102.3	96.6	101.7
44'6	9	99.7	101.2	100.4	99.7	93.3	119.3	102.7	139.2	102.8	97.1	101.6
44'66'	10	97.7	107.6	107.3	100.3	94.6	123.2	101.0	135.3	102.4	96.5	100.9
44'6	11	94.3	102.1	100.6	98.2	93.3	79.9	88.2	-	108.0	100.6	105.6
44'5'6	12	89.8	129.5	122.7	98.0	90.0	154.5	-	-	102.6	95.3	100.4
33'44	13	102.7	102.2	100.7	99.5	71.8	104.8	62.8	-	100.5	94.7	98.2
al mean overy %		98.8	102.5	102.9	97.9	93.9	120.5	99.0	138.4	104.6	97.5	103.0

results are in % of recovery

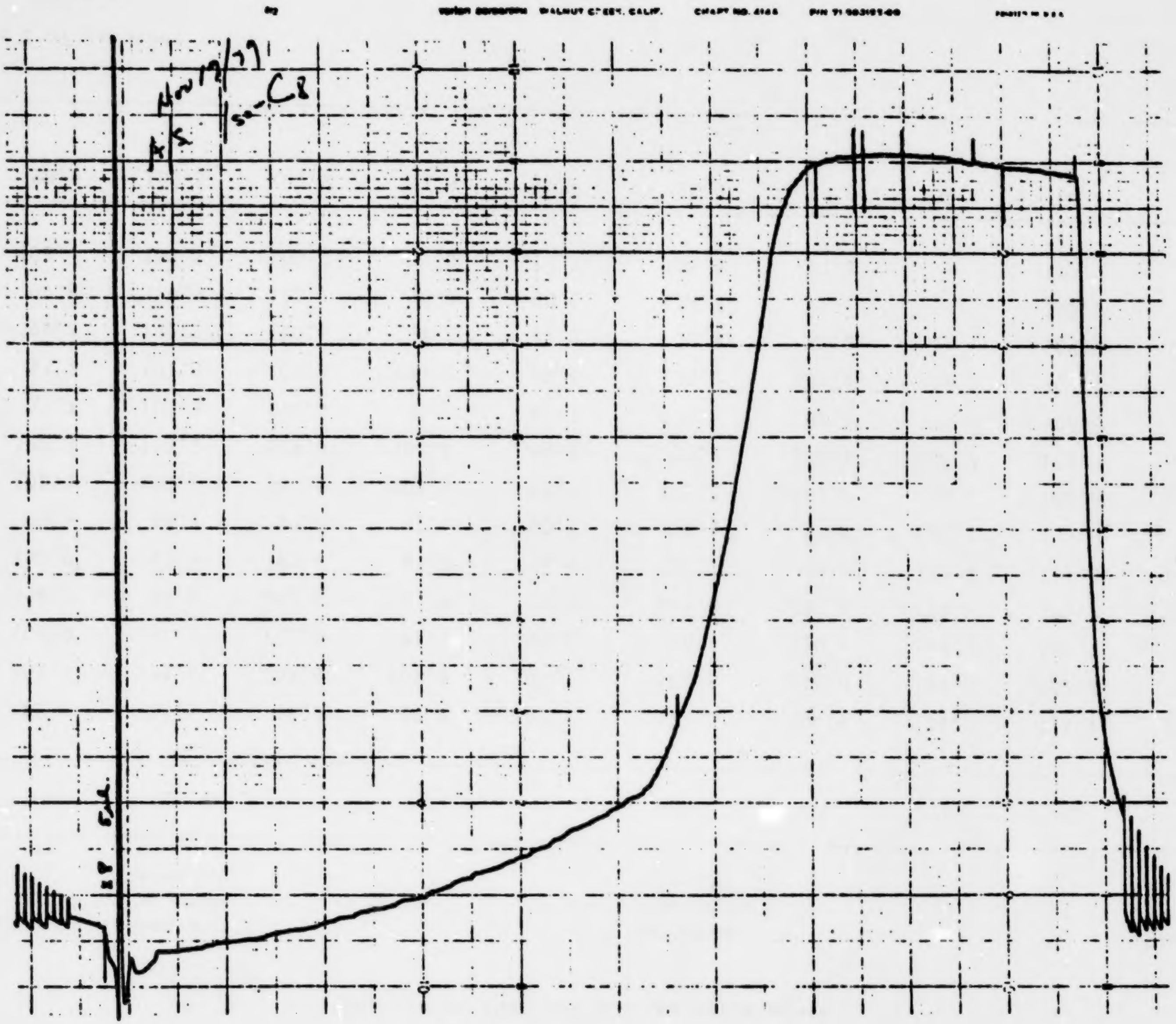


Figure 1. Automated injection of blank solvent.

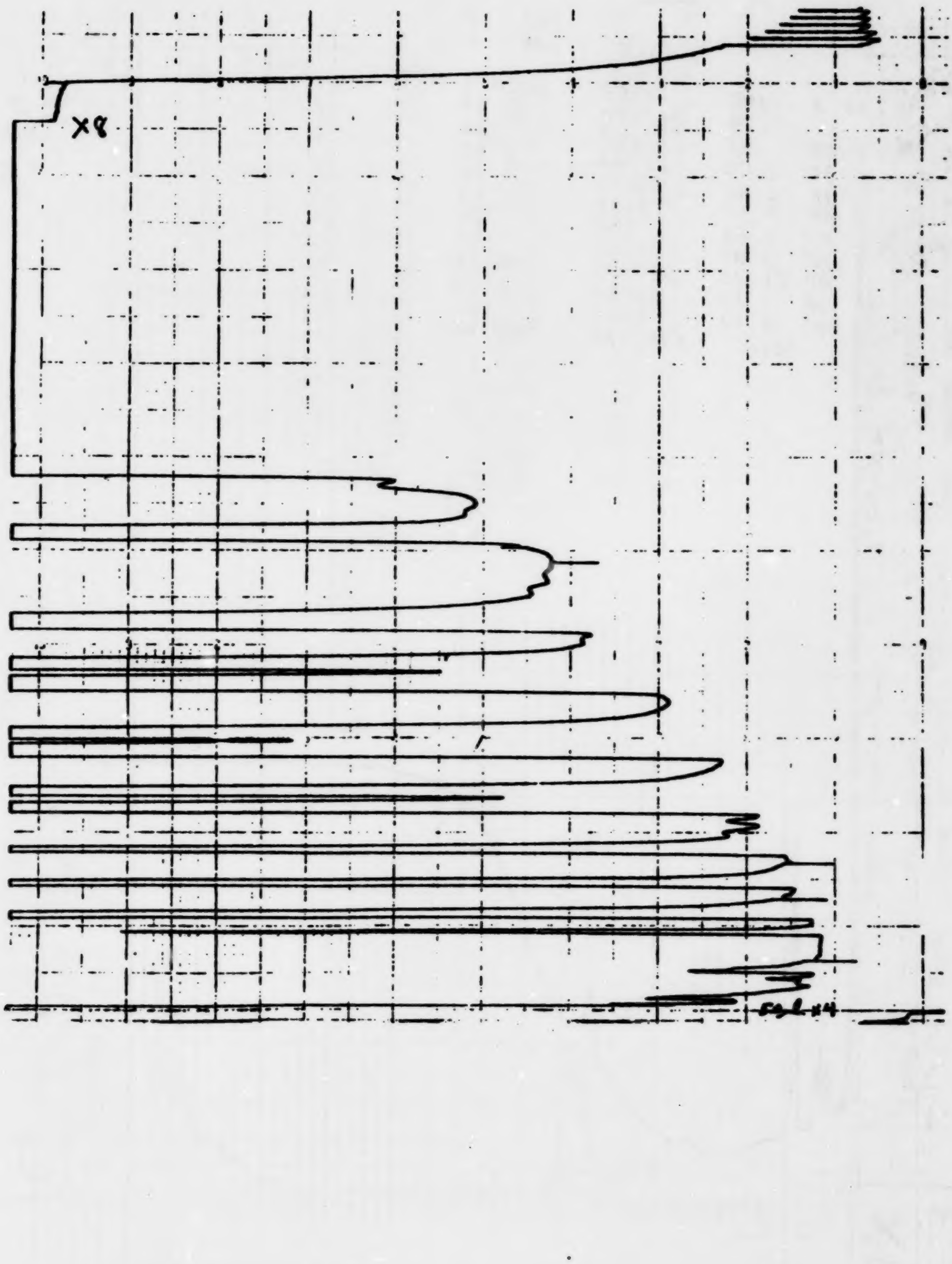
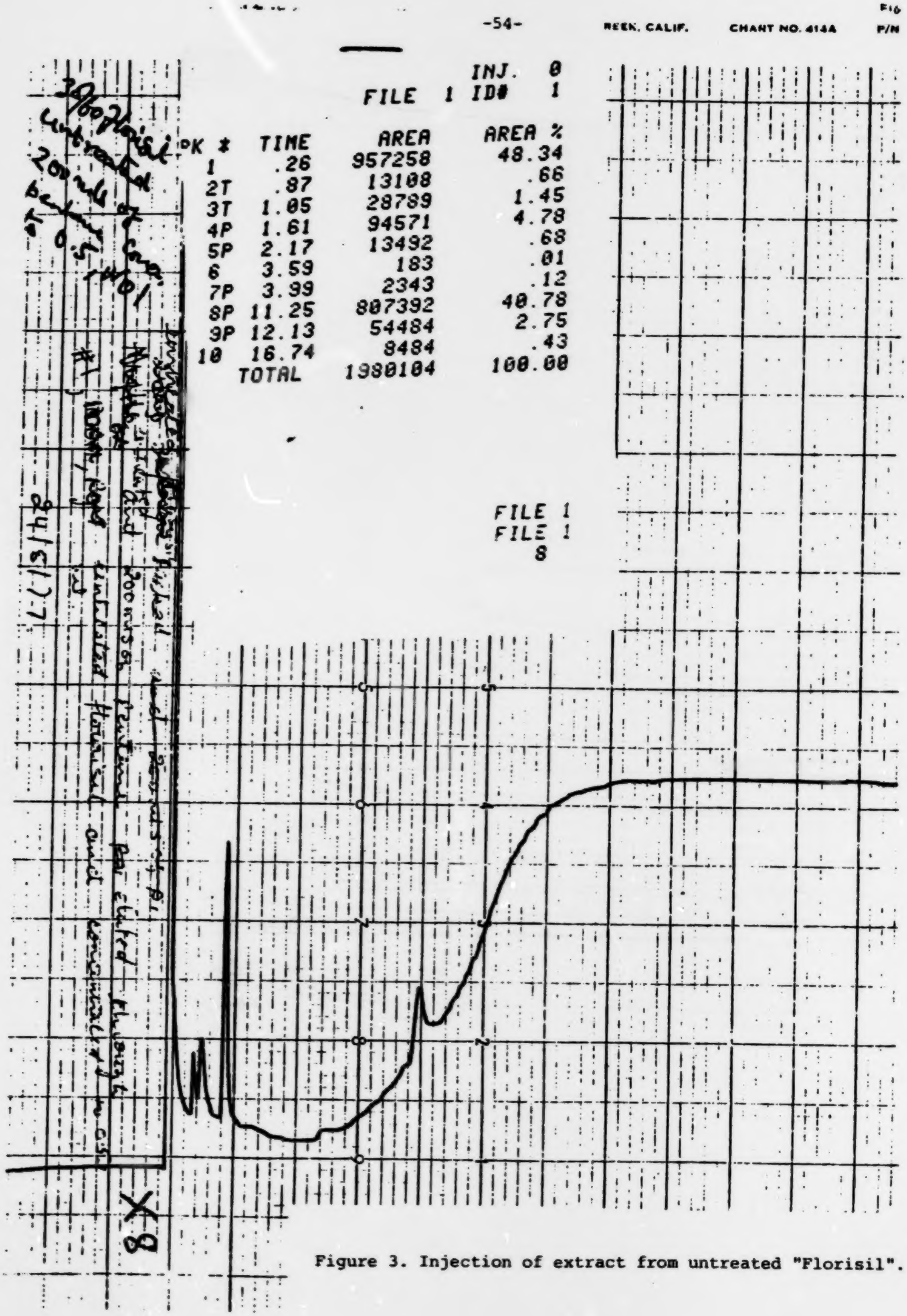


Figure 2. Automated injection of PCB standard preceeding injection of blank solvent.



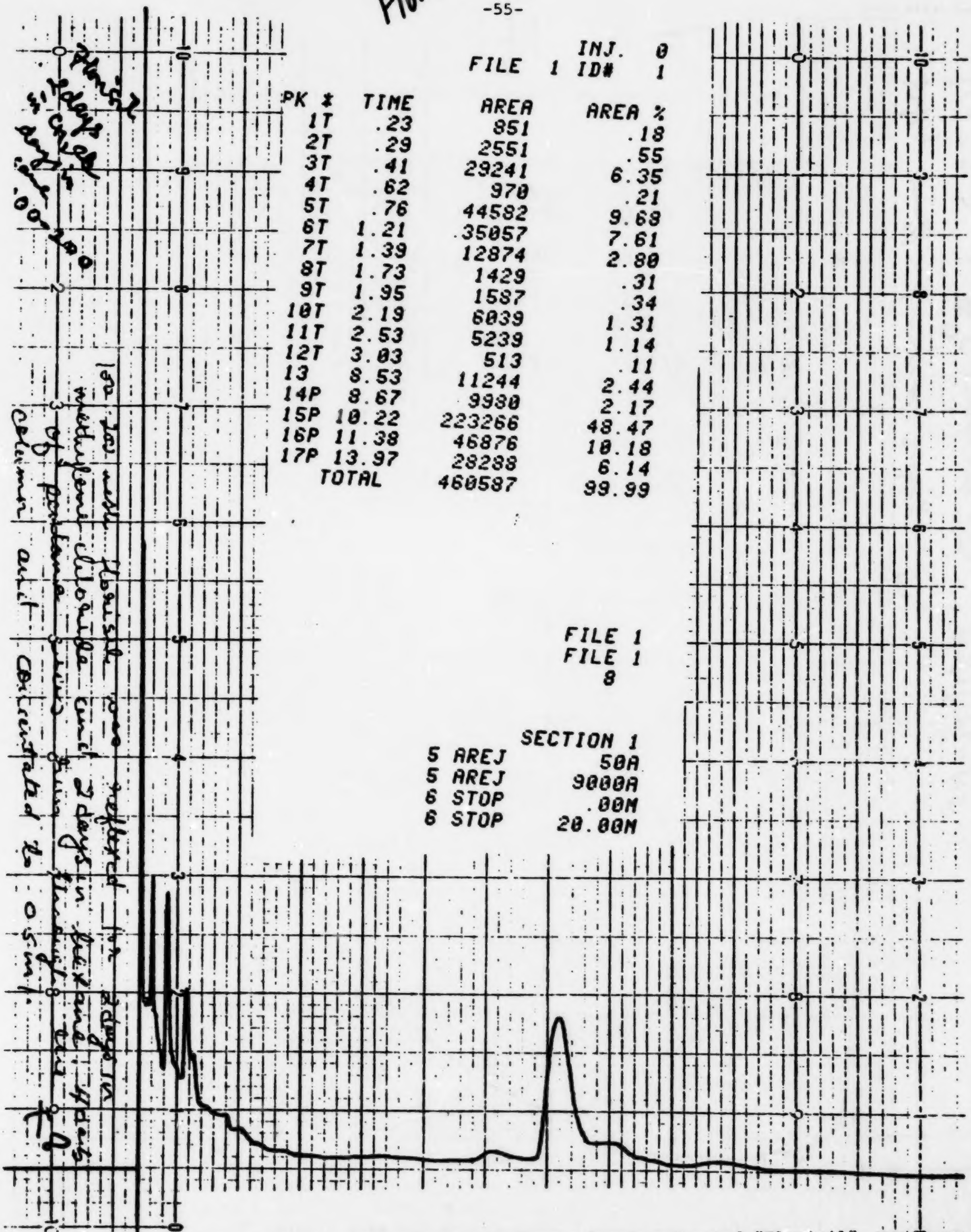
```
                          INJ.   0
                FILE   1  ID#    1

PK #   TIME       AREA       AREA %
1       .26     957258       48.34
2T      .87      13108         .66
3T     1.05      28789        1.45
4P     1.61      94571        4.78
5P     2.17      13492         .68
6      3.59        183         .01
7P     3.99       2343         .12
8P    11.25     807392       40.78
9P    12.13      54484        2.75
10    16.74       8484         .43
      TOTAL    1980104      100.00


                          FILE 1
                          FILE 1
                               8
```

3% of Florisil untreated
200 ml of conc.
benzene 0.5 µl

Injected [illegible] flushed and [illegible]
[illegible] and 200 ml of [illegible] eluted [illegible]
#1 [illegible] untreated Florisil and concentrated to 0.5
24/5/77

Figure 3. Injection of extract from untreated "Florisil".

Florisil



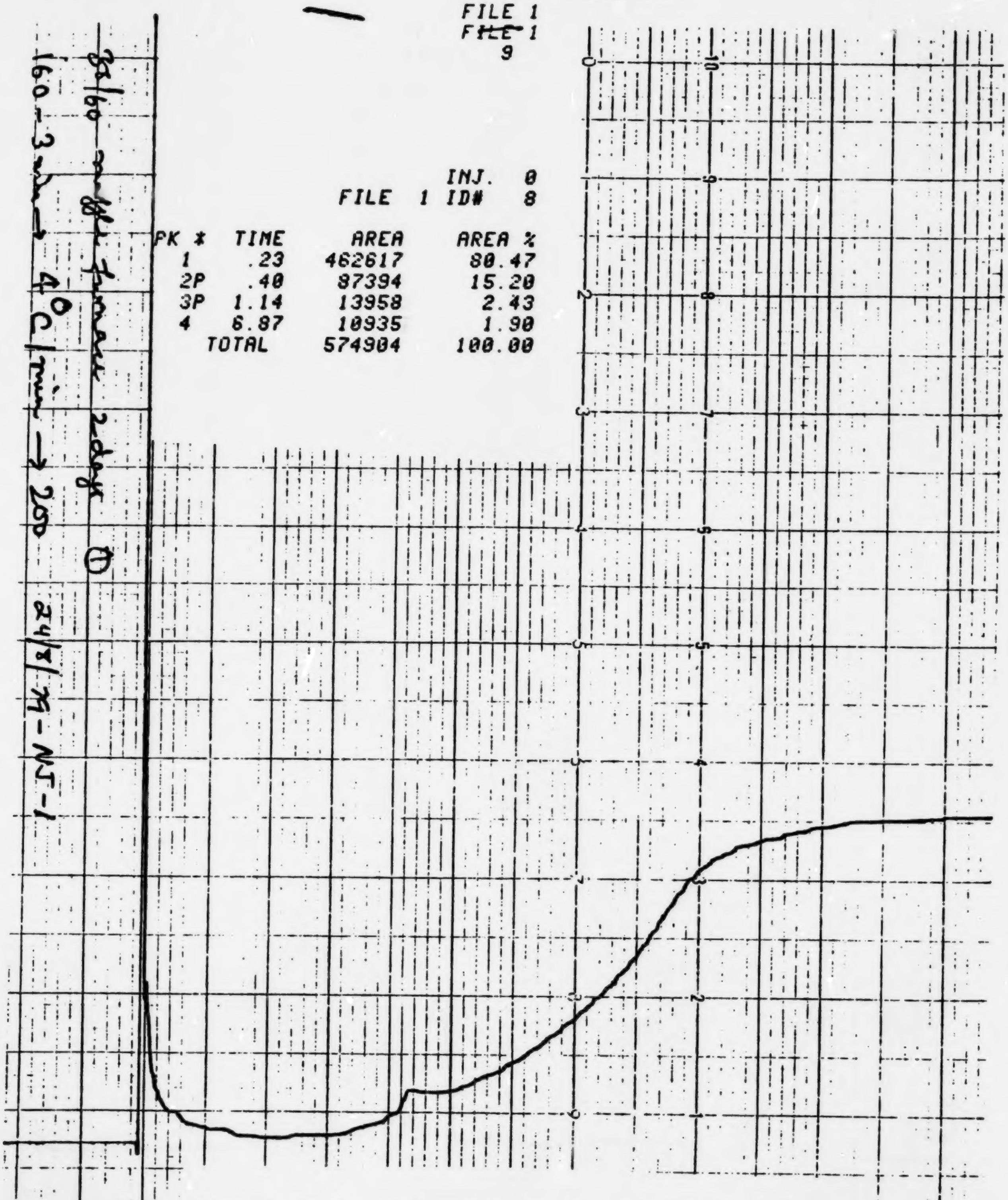
```
                              INJ.   0
                    FILE   1  ID#    1
PK #    TIME        AREA      AREA %
  1T     .23         851         .18
  2T     .29        2551         .55
  3T     .41       29241        6.35
  4T     .62         970         .21
  5T     .76       44582        9.68
  6T    1.21       35057        7.61
  7T    1.39       12874        2.80
  8T    1.73        1429         .31
  9T    1.95        1587         .34
 10T    2.19        6039        1.31
 11T    2.53        5239        1.14
 12T    3.03         513         .11
 13     8.53       11244        2.44
 14P    8.67        9930        2.17
 15P   10.22      223266       48.47
 16P   11.38       46876       10.18
 17P   13.97       28288        6.14
       TOTAL      460587       99.99


                              FILE 1
                              FILE 1
                                   8


                          SECTION 1
              5 AREJ            50A
              5 AREJ          9000A
              6 STOP           .00M
              6 STOP         20.00M
```

Florisil 2 days in CH2Cl2 2 days in hexane .00-200

10g 200 mesh florisil was refluxed for 2 days in methylene chloride and 2 days in hexane. 4 mls of pentane was run through the column and concentrated to 0.5 ml.

×8

Figure 4. Injection of extract from solvent extracted "Florisil".



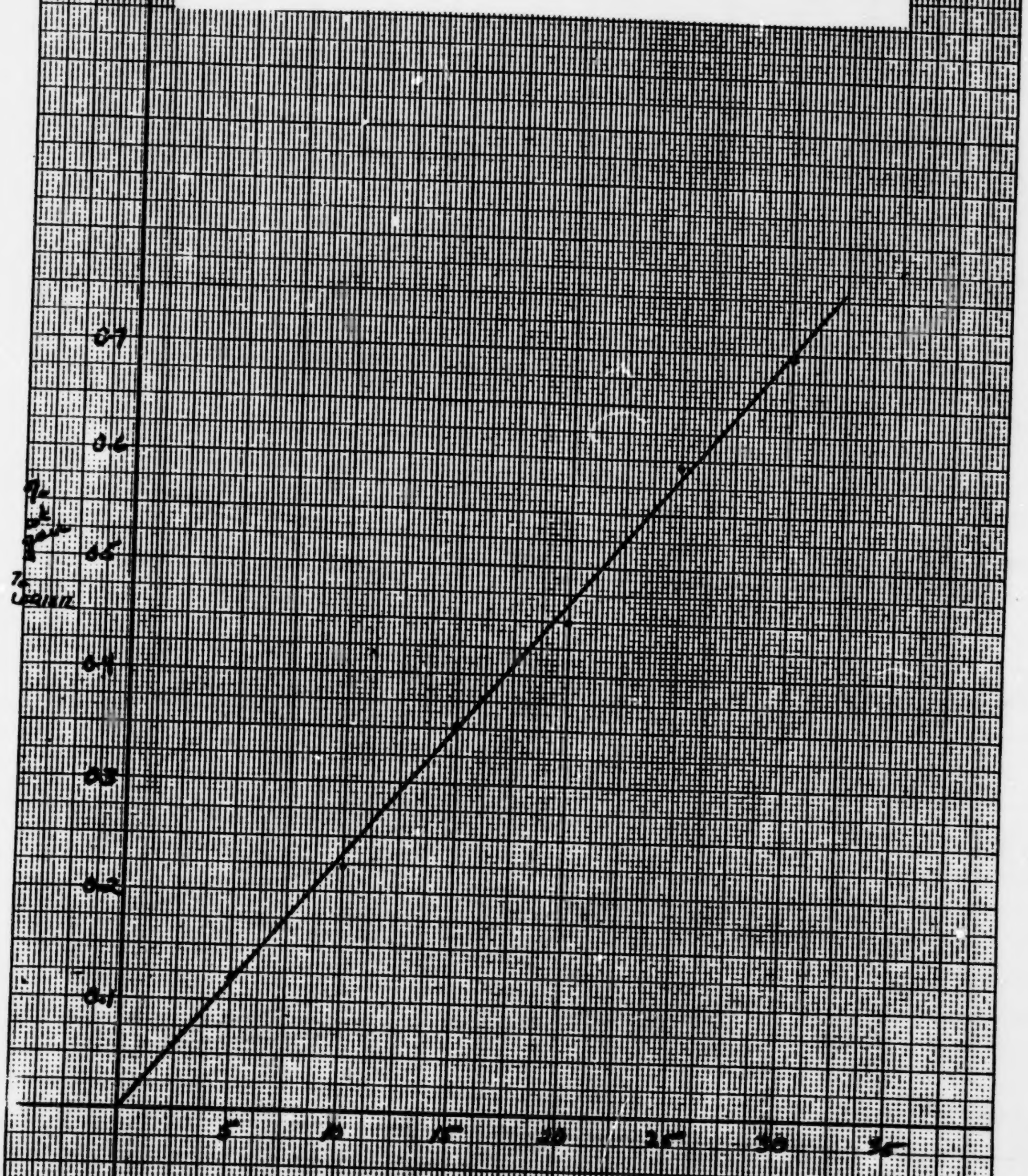
```
FILE 1
F~~ILE~~ 1
     9

              INJ.   0
      FILE  1 ID#    8

PK #  TIME     AREA    AREA %
 1     .23   462617     80.47
 2P    .40    87394     15.20
 3P   1.14    13958      2.43
 4    6.87    10935      1.90
  TOTAL      574904    100.00
```

30/60 mesh after furnace 2 days ①

160 - 3 min → 4°C/min → 200   24/8/79-NJ-1

Figure 5. Injection of extract from thermaly treated "Florisil".

Figure 6. Correlation between "Florisil" weight gain and time.



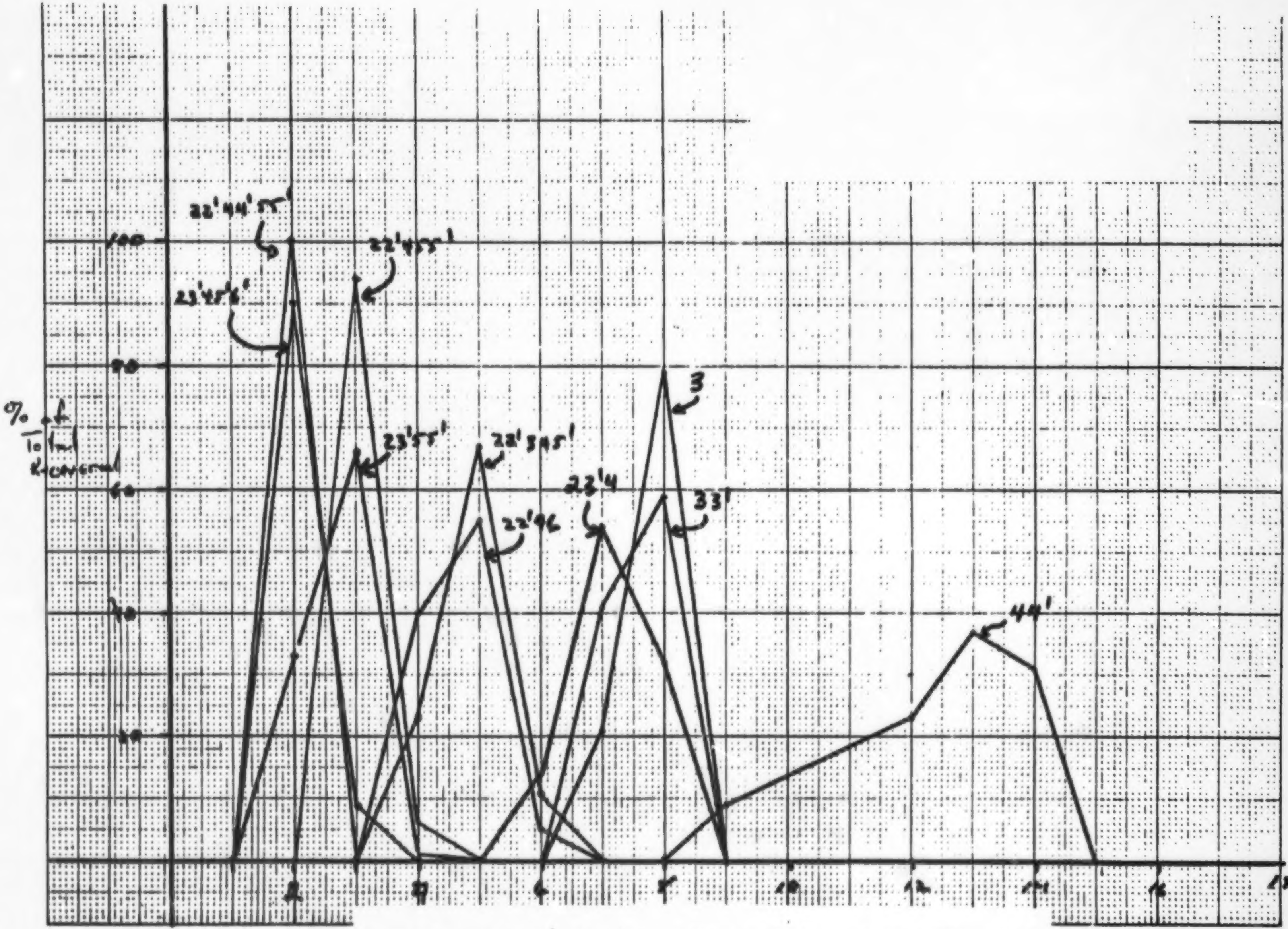


Figure 7. PCB isomer elution profile on fully activated "Florisil".

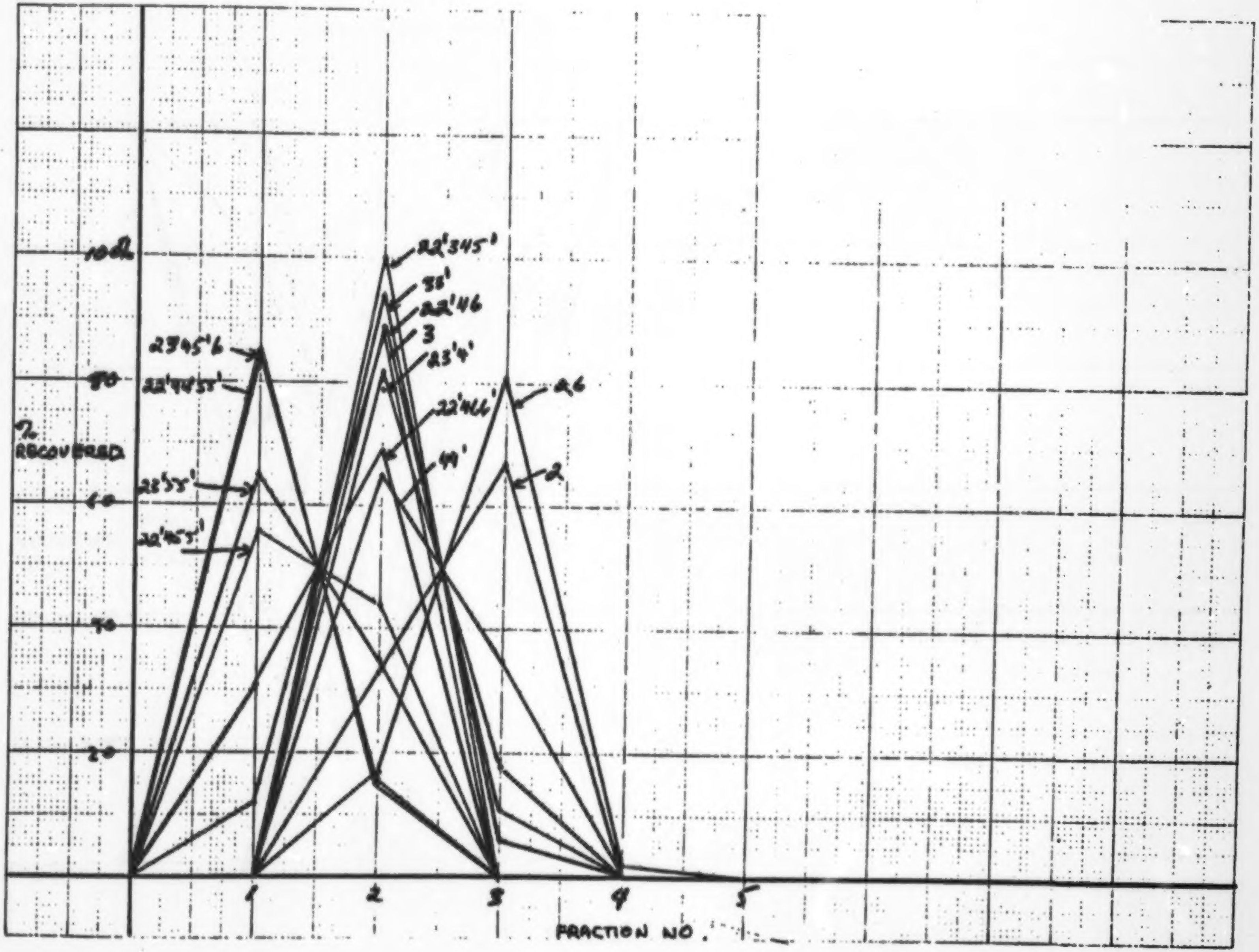


Figure 8. PCB isomer elution profile on 1% water deactvated "Florisil".

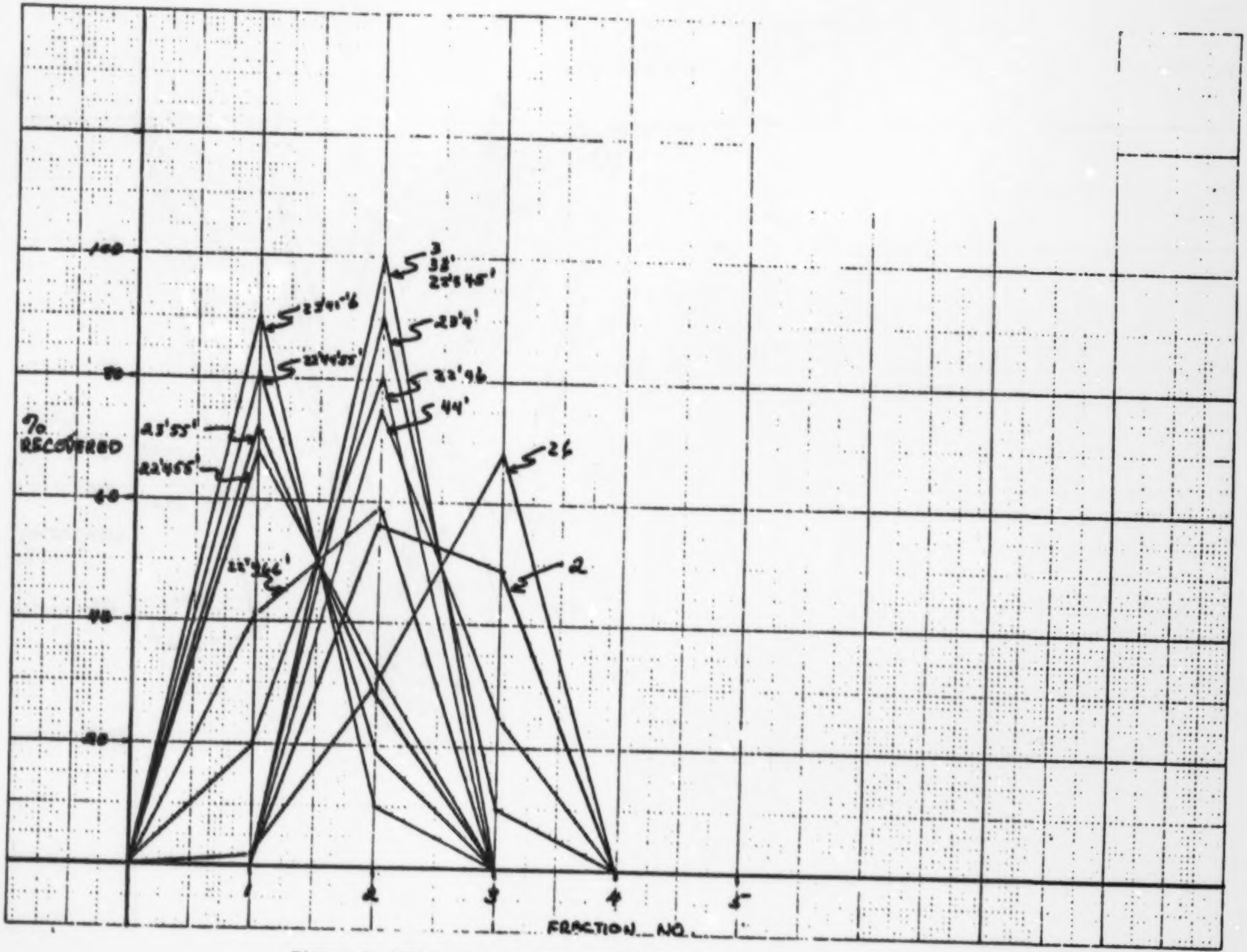


Figure 9. PCB isomer elution profile on 2% water deactivated "Florisil".

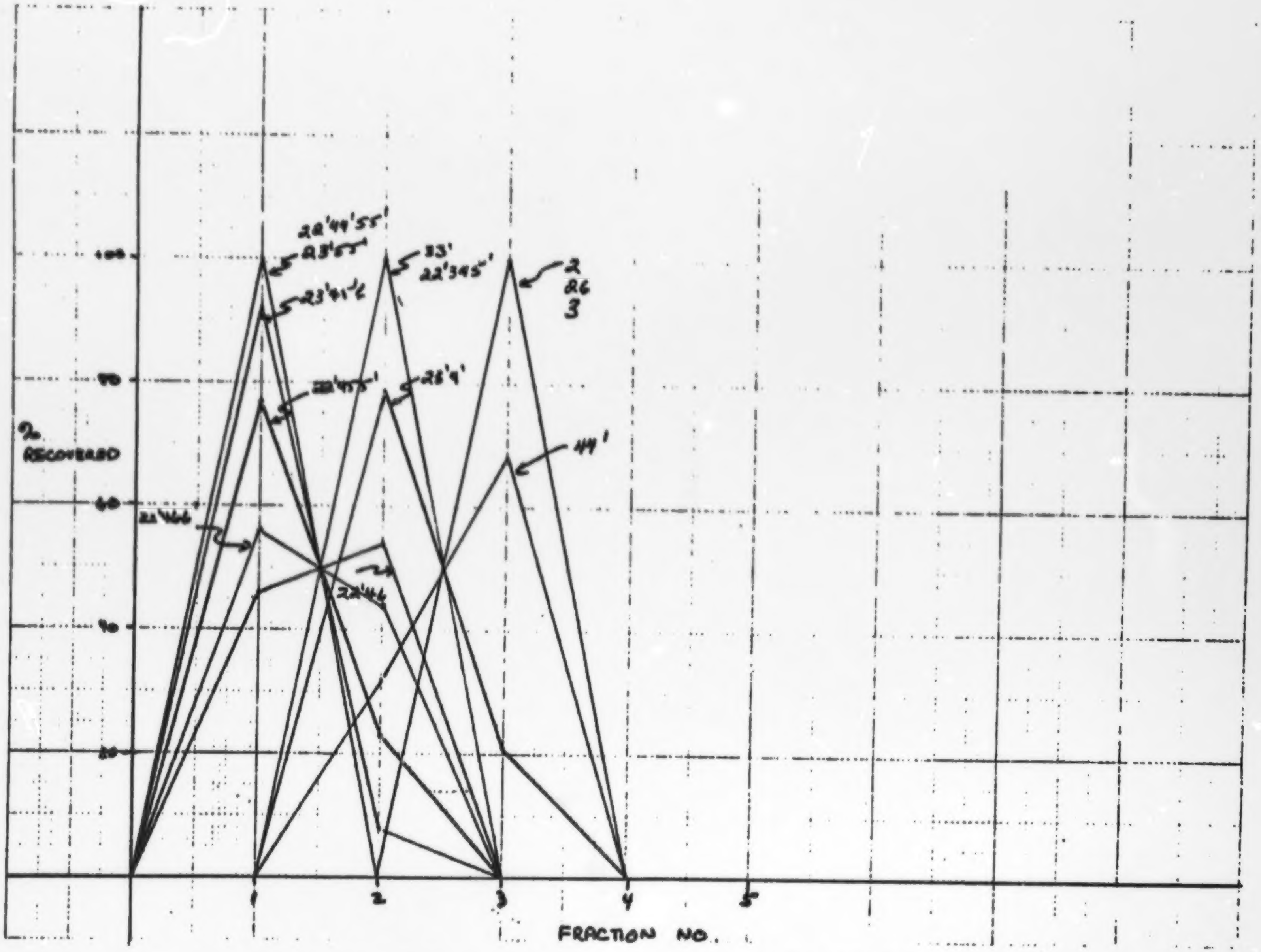


Figure 10. PCB isomer elution profile on 3% water deactivated "Florisil".

APPENDIX A

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Definition of Responsibilities of Personnel
Detailed Protocol for Ambient Air Analyses of PCB's
Detailed Quality Control Program for Ambient Air Analysis of PCB's

MANPOWER REQUIREMENTS

Function	No.
Project Principal	1
Technical Director	1
Group Leader	2
Chemist	2
Technician	2

PROJECT PRINCIPAL: FUNCTIONS

- overall organization, administration
- liaison with other contract participants (i.e. IEC and Moniteq)
- provision of resources
- administrative liaison with MOE
- report overview, proofreading and final approval
- scheduling and approval of operator shift assignments
- overall monitoring of contract and schedule compliance
- overall performance evaluation of all personnel associated with the project
- approval of all supplies purchasing related to the project.

TECHNICAL DIRECTOR: FUNCTIONS

- technical liaison with MOE
- technical goal setting and task definition
- report organization and writing
- data collation and filing
- schedule setting
- inspection of incoming cartridges
- quality control audit and liaison with IEC for Independent audit
- procedure writing and approval
- prompt identification and definition of problems to ensure their resolution
- weekly progress reports to PROJECT PRINCIPAL
- all record keeping procedures and their adequacy
- technical liaison with IEC regarding meteorological data reduction and reporting
- ordering of supplies to fulfill schedule commitments after approval by PROJECT PRINCIPAL
- ensure that clerical tasks relating to sample reception are completed adequately
- authorization of procedural changes and provision of written revisions to all operational lab staff
- assist in any manner deemed necessary by the PROJECT PRINCIPAL.

GROUP LEADERS: FUNCTIONS

- lab organization and supervision of chemists and technicians to ensure most efficient utilization of their time for the achievement of all operational tasks of the project.
- inventory control and order consumable supplies
- equipment operation, maintenance, performance checks and supplier liaison, including daily equipment QC and sample analysis
- cartridge preparation, proving, packaging and shipping
- data reduction calculations, and tabulation
- assist in report writing as required by TECHNICAL DIRECTOR
- ensure that all laboratory operations and techniques are followed as consistently as possible by all operators over both shifts
- bring all operational or administrative problems directly to the attention of the TECHNICAL DIRECTOR AND/OR PROJECT PRINCIPAL to ensure their prompt resolution, including recommendations for procedural changes
- efficient and proper completion of shift assignments to ensure availability of glassware for following day's or shift's analysis
- approval of proving analyses of solvents, glassware, "Florisil", cartridges and cartridge blanks
- ensure that the pre-clean-up fraction of each sample is sealed in a 10 ml ampoule each day, for subsequent delivery to MOE
- ensure that the remaining sample after GLC analysis is promptly sealed in a 1.0 ml ampoule for subsequent delivery to MOE
- assist in any manner deemed necessary by the TECHNICAL DIRECTOR or PROJECT PRINCIPAL.

CHEMISTS: FUNCTIONS

- ensure all defined procedures and techniques are followed by himself and the corresponding shift technician in all aspects of glassware proving, cartridge preparation and proving, and sample analyses
- acts as back-up operator for GC analysis and calculation
- write-up of raw data pertaining to all preparatory work
- assist in calculations and/or report writing as required by GROUP LEADER
- keep laboratory work areas clean, organized and orderly at all times
- training of back-up technician in all preparatory procedures
- assist in any manner deemed necessary by the GROUP LEADER or higher project personnel
- follow defined procedures in all aspects of glassware, sample and cartridge preparation
- keep laboratory work areas clean, organized and orderly at all times
- assist in any manner deemed necessary by the GROUP LEADER, or higher project personnel.

DETAILED PROTOCOL FOR AMBIENT AIR ANALYSES OF PCB's

I. General Glassware Cleaning, Proving and Maintenance Procedure

II. Procedure for Cleaning and Proving of "Florisil"

III. Procedure for Packing of "Florisil" Cartridges: Their Proving, Reactivation and Maintenance

IV. Instructions for Packaging and Shipment of Sampling Cartridges

V. Instructions for Sample Reception and Log In

VI. Extraction and Clean-Up of Field Samples

VII. Instrumental Analyses - Daily and Weekly Checks and Operating Procedures

VIII. Procedures for Calculations of Analytical Results

I: GENERAL GLASSWARE CLEANING, PROVING & MAINTENANCE PROCEDURE

*All new glassware, prior to useage in trace analysis of PCB's will be cleaned by the following procedure.

*Under no circumstance is the glassware to be handled with bare hands. Use clean cotton gloves.

*Solvents used in this procedure must be distilled in glass, suitable for pesticide residue analysis. Handle all glassware with clean cotton gloves or precleaned metal tongs.

1) Utilizing soft brushes to minimize scratching, all new glassware will be soaked in and scrubbed with hot laboratory detergent, rinsed with tap water, then distilled water.

2) Soak glassware in a hot solution of 2% "Decon 75" in distilled or demineralized water overnight (at least 12 hours). This will be repeated whenever deemed necessary (at least once weekly). Completely rinse with distilled water.

*Glassware being cycled for reuse may be reproven with the following steps.

3) Rinse each piece with at least 25 ml of acetone, then 25 ml of hexane. Bake all glassware at 250°C for at least 16 hours in an oven dedicated to this purpose only.

4) Glassware batches (lots of 15) used for preparation of a sample lot (15) will be rinsed with pentane after cooling. Two rinses with pentane will be used. The first rinse of 25 ml/piece will be discarded as waste. The second rinse of 10 ml/piece will be collected into one 500 ml "Kuderna-Danish" evaporator to which 1.0 ml of iso-octane is added, evaporated to 1.0 mls and subsequently analyzed by GC.

Based on the result of this analysis and a predetermined threshold, the batch of glassware will be accepted for use or rejected, requiring re-cleaning beginning at step 2.

5) Glassware which has been rinsed with pentane should be stored at 135°C in an oven designated for the purpose until the proving analysis is complete. Immediately prior to use, the proven glassware is removed from the storage oven and given a rinse with pentane which is discarded.

REQUIRED GLASSWARE BATCH FOR 15 SAMPLES (PROVEN CLEAN)

15 - elution reservoirs for sample recovery
15 - 500 ml capacity "Kuderna-Danish" Evaporators with 10 ml "Kontes" receivers
1 - 250 ml graduated cylinders
15 - smooth watch glass covers, 50 mm dia.
1 - 25 ml graduated cylinder
15 - micro "Snyder" column
15 - "Florisil" elution columns
1 - 100 ml beaker
15 - 50 ml calibrated centrifuge tubes with ₸ 19/22 joints
1 - 50 ml graduated cylinder
15 - 5 ml volumetric pipets
15 - 10 ml "Kontes" receivers (may be same as above)
1 - glass rod 4 mm dia. 60 cm length
1 - powder funnel 4" dia.

*also rquired but not batch proven along with the glassware batch -but proven nonetheless.

- clean aluminum foil to cover items not in use
- glass wool
- boiling stones
- "Florisil" 100/200 mesh 50 grams
- "Pasteur" pipets (90)
- sample vials for GC analysis (15) 1.0 ml cap.
- glass wash bottles to contain nanograde solvents
15 - 10 ml glass ampoules
15 - 1.0 ml glass ampoules

II: PROCEDURE FOR CLEANING AND PROVING OF "FLORISIL"

* All solvents used in this procedure must be distilled in glass, suitable for pesticide residue analyses.

* Handle all glassware with clean cotton gloves or pre-cleaned metal tongs.

Two mesh sizes of "Florisil" are being used:

1) 30/60 mesh is used for the sampling cartridges
2) 100/200 mesh is used for the elution clean-up column

As supplied by the manufacturer, "Florisil" requires considerable clean-up, before being suitable for use. "Florisil" is cleaned by thermal desorption of volatile organics, and combustion of absorbed organics at high temperatures - a tube furnace is sued.

1) A quantity of "Florisil", approx. 500 grams, is placed into the combustion tube which has been precleaned by preheating in the tube furnace. Glass wool is used to hold the "Florisil" in place. The packed length of "Florisil" must not exceed the heated length of the tube furnace (35 cm).

2) A flow approx. 1.0 l/min of "Florisil" filtered air is passed through the tube. The furnace is slowly heated up to 650°C at the rate of approx. 100°C per hour, and maintained were for 24 hours.

3) After 24 hours the air flow is decreased and the tube and furnace allowed to cool to room temperature.

4) The "Florisil" is removed from the tube, transferred into a clean storage bottle or beaker and placed in the "Florisil" storage oven at 135°C until tested.

5) If proven unsuitable the remaining sample should be replaced into the tube and given further 24 hours of cleaning, although

the slow temperature raise would not be needed to bring the sample to 650°C.

"FLORISIL" PROVING

1) "Florisil" will be proven by placing the material in one of the appropriate pieces of glassware i.e. - a sampling cartridge (for 30/60 mesh) or - an elution clean-up column for 100/200 mesh using the procedures below:

2) 30/60 Mesh "Florisil".

Prepare a sampling cartridge as follows. A small plug of glass wool is placed in the tube and gently packed to 0.5 cm. Using a funnel, "Florisil" is slowly poured into the tube to a bed height of 5.0 cm. The tube is gently tapped on the bottom to settle the packing, and additional "Florisil" added to bring the bed to 5.0 cm. An additional 0.5 cm bed of glass wool is added on top.

Place a cooled elution reservoir into a sealing cap, and insert to the level 1.0 cm above the glass wool. Tighten the screw cap to seal, and set in a rack with a watch glass covering the reservoir. Rinse the tip of the sample tube with nanograde acetone than nanograde pentane to eliminate any external contamination, into a waste beaker; elute with two (2) solvents*, first - 200 ml 10% methylene chloride in pentane which is collected in a waste beaker and second with 200 ml pentane of which the first 170 ml are collected in a waste beaker. The last 30 ml of pentane are collected for analysis, using a 50 ml graduated collection tube. Add 1.0 ml of iso-octane as keeper, place a micro "Snyder" column on top, reduce volume to 1.0 ml and submit for GC analysis. Based on this analysis, the "Florisil" batch will be either accepted for use, or rejected.

*Ensure that the solvent level in the cartridge tube does not rise continuously upwards. If it does, this indicates a leak in the screw cap seal, and this should be tightened immediately, preventing further rise of the solvent. Solvent reaching the seal may contaminate the sample and invalidate the "Florisil" proving; Note Discrepancy.

3) 100/200 Mesh "Florisil"

Prepare a clean-up column as folows:

Set up an elution column with the intergral solvent reservoir in its rack. Asemble the chromatographic column by inserting a 0.5 cm glass wool plug in the bottom. Remove a small "Florisil" sample from storage oven, leave "Florisil" open to atmosphere and cooling. Place 50 ml centrifuge collection tube under the column. Pour "Florisil" from beaker into column while tapping the column to form a bed 22 cm high.

Add pentane, 1½ ml at a time, until entire column is wetted. Slowly add 40 ml pentane to reservoir and slowly elute. Collect 25 ml pentane in the 50 ml centrifuge collection tube. Add 1.0 ml of iso-octane as keeper, place a micro "Snyder" column on tip, add a boiling stone and reduce volume to 1.0 ml and submit for GC analysis. Based on this analysis the "Florisil" batch will be either accepted for use or rejected.

4) Preparation of 100/200 Mesh "Florisil"

"Florisil" which has been proven suitably clean needs to be deactivated prior to use to ensure complete recovery of all PCB isomers.

"Florisil" is placed on a large proven glass bottle (of sufficient size to contain one week's requirements). The "Florisil" is deactivated by addition of 3% (by weight) of distilled, deionized water (HPLC grade water would be suitable).

The mixture is sealed tightly and slowly tumbled for twenty-four hours. After thorough tumbling the "Florisil" is subdivided into smaller clean bottles (4 oz.) with aluminum lined caps. Each bottle should contain sufficient "Florisil" for the days work only. Any excess, should be reactivated before use, as previously described.

III. PROCEDURE FOR PACKING OF "FLORISIL" CARTRIDGES: THEIR PROVING, REACTIVATION AND MAINTENANCE

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*All solvents used in this procedure must be distilled in glass, suitable for pesticide residue analysis.

*Handle all glassware with clean cotton gloves or precleaned metal tongs.

1) All sampling cartridges will be dissassembled and glass components will be cleaned prior to use following steps 2 - 6 of the General Glassware Cleaning Proving and Maintenance Procedure (pg. A 7). A lot size of 50 is recommended.

2) "Florisil" 30/60 mesh, approximately 4 g/cartridge, is required from a proven stock stored at 135°C until required. Refer to "Procedure for Cleaning and Proving of Florisil" (pg. A 10).

   Glass wool is also required cleaned by "Soxhlet" extraction in methylene chloride, then dried in an oven at 135°C. The wool is then extracted in pentane and the extract concentrated in a "Kuderna-Danish" evaporator and analysed by GC. The glass wool should remain at all times in its extraction thimble and be ultimately stored in a proven clean bottle to eliminate unnecessary transfer steps.

3) Prepare cartridges as follows. (A lot of 5 batches of 10 are recommended). Set cartridges vertically in a rack. A precleaned stainless steel screen is inserted into the tube by using a pair of tweezers (which have been cleaned with solvent). The screen is placed in position at the bottom of the tube by using a glass rod. A small plug of glass wool is placed in the tube and gently packed to 0.5 cm. Using a funnel, "Florisil" is slowly poured into the cartridge to a bed height of 5.0 cm. The cartridge is gently tapped on the bottom to settle the packing, and additional

"Florisil" added to bring the bed to 5.0 cm. An additional 0.5 cm bed of glass wool is added on top. Assemble the glass foot and cap, having previously wiped the "Teflon" surfaces only of the the sealing ring with pentane on a clean cotton cloth. Store vertically in large glass beakers until proving.

4) All prepared cartridges will be given a final clean-up and proving by solvent elution as described. Set-up elution reservoirs and proceed as in steps 2, 3, 4 of the section entitled "Extraction & Clean Up of Collected Samples" (pg. A 21) noting the following exceptions:

(i) no "Teflon" tube plug has been used up to this point
(ii) elution will occur with two (2) solvents

- first 200 ml 10% methylene chloride/pentane which is collected in a waste beaker

- second 200 ml pentane of which the first 170 ml are collected in a waste beaker

(iii) the last 30 ml of pentane are collected from each of the batch of ten and combined for analysis. The combined extracts of 300 ml are evaporated in a "Kuderna-Danish" with 1.0 ml of iso-octane as keeper. The volume is reduced to 1.0 ml and submited for GC analysis. Based on this analysis, the batch of cartridges analyzed will be either accepted for use, or rejected as requiring complete recleaning and repacking.

5) Plastic caps will undergo no cleaning prior to use. "Teflon" sealing rings will be carefully wiped on the "Teflon" surfaces only using a cotton cloth and pentane.

6) Prepared and proven sampling cartridges will be reactivated as follows: the bulk of the residual pentane will be removed by a gentle stream of "Florisil" filtered air (a cartridge may conveniently be used for this). Using cotton gloves, the cap is removed from

the cartridge, and the glass foot removed from the sealing ring using tweezers (store on a clean piece of aluminum foil). Replace the cap and sealing ring onto the cartridge and place on the air line manifold and tighten into place. When the manifold has its compliment of cartridges (5-10), the air is passed through the cartridges until they are visibly dry. The air flow is stopped and the cartridges with sealing rings are removed from the manifold. Screw caps are removed and reassembled with a glass foot using tweezers and stored in a clean covered beaker until required for shipment of the tubes. Use of cotton gloves is recommended while assembling of screw caps to prevent contamination of the parts with skin oils.

Cartridges are placed vertically in large beakers and placed in an oven at 135°C which is dedicated to the purpose of maintaining active "Florisil". A minimum of two days is required for reactivation. Cartridges will remain in this oven until required for shipment.

IV. INSTRUCTIONS FOR SHIPMENT AND PACKING OF CARTRIDGES

* Handle all glassware with clean cotton gloves or precleaned metal tongs.

* Solvents used in this procedure must be distilled in glass, suitable for pesticide residue analysis.

1) Sampling cartridges to be shipped will have been proven and a blank chromatogram available. The cartridges will have been reactivated at 135°C in an oven dedicated to "Florisil" storage for at least 2 days, and should be ready at least one full day in advance of the anticipated date of shipment.

2) Each batch of 10 cartridges must have a blank analysis performed on one tube from the batch. This is performed by following steps 2 to 7 of the procedure entitled "Extraction and Clean'up of Collected Samples" (pg.) noting the following exception.

   - that no screw cap, glass foot or sealing tube has been used at this point

3) Prepare tube seals for the sampling cartridges, if not already available. Fluorinated polypropylene tubing (¼" OD x .030" wall thickness) is used. The tubing should be cleaned by rinsing the inside of a suitable length of the tube with hexane, after which a further rinse with pentane (200 ml) is collected, evaporated and analyzed to prove that the tubing is suitably clean for use.

   Using a heat gun to soften the FEP tubing (it will become more transluscent), form the tubing on the 6mm end piece of a sampling cartridge. Set the FEP tubing onto the glass a minimum of ¼". Cut the tubing 1" from the end of the glass. Heat the end of the FED tube until clear and pinch between the jaws of a pair of warm pliers. Reheat and pinch again 90° to the previous step. Allow the tubing to cool fully before attempting to remove.

Use a knife to cut off any excess material around the glass tube which has "buckled" or deformed. Remove the seal by gently grasping the FEP tubing over the glass with the jaws of the pliers and "twist" until it breaks loose. The seal can now be easily removed by hand by pulling the flattened end

4) Prepare sample identification tags for the cartridges to be shipped by writing the appropriate blank analysis and proving analysis identification numbers and the date of packing on the tag.

5) Prepare a sufficient number of sealing caps with the glass foot installed. Handle the glass foot (after cooling) with cotton gloves only.

6) Remove the sample cartridges from the storage oven and cool sufficiently to permit handling. Place a prepared FEP tube seal onto the cartridge, and then an assembled glass foot, sealing ring, screw cap combination. Push the glass foot down to hold the glass wool and "Florisil" in place during shipping. Twist the wire of the sample identification tag around the neck of the tube just under the sealing cap, so that the tag is fully secured to the tube.

7) Place the prepared tubes in their individual glass shipping containers (along with precleaned aluminum foil) and ensure that the cartridge is immobile in the cylinder - Pack with glass wool or cotton if necessary.

8) Pack the containers into the shipping cartons and place in one copy of a log sheet, a set of instructions for the field crew, and an envelope containing the sampling data sheets.

V. INSTRUCTIONS FOR SAMPLE RECEPTION AND LOG-IN

Samples will be received from a courier in large boxes.

1) On receipt of the sample box observe external packaging for signs of gross mishandling and report to the project manager for further action.

2) Open container and inspect packing slip. Observe shipping date, and confirm identity of sample containers to corresponds to the packing slips.

   The identification code will consist of a multi digit code indicating the sampling region, the sampling phase, the specific site and a number identifying the sample specifically, e.g. THU-S-UI-1

3) Prepare the work traveller sheet for each sample tube received by:
   - typing the sample identification on each sheet
   - typing the date of receipt of each sheet

4) Type two (2) sets of gummed self-adhesive labels with sample identification code. Confirm that no mistakes are present on these gummed labels.

5) Samples should be placed in the office of the project manager for distribution to the analyst(s).

6) Under the supervision of the project manager the sample tubes will be removed from their individual shipping containers. The tubes will be inspected to ensure that the seals are intact i.e. the screw cap seal was tight and the glass foot in place, and that the FEP tubing seal is in place. Any discrepancies will be reported in the appropriate place on the work traveller sheet.

7) The cartridge blank (CB-) number will be recorded on the work traveller sheet. Ensure that the stub portion of the sample identification tag has the correct sample identification code on it. Cut the tag at the indicated line and staple the tag to the work traveller sheet in the indicated place.

VI. EXTRACTION AND CLEAN UP OF COLLECTED SAMPLES

* Lots of 15 sample cartridges will be most convenient to work with. Before proceeding insure that a proven glassware batch is available for use, as described in the general glassware cleaning and proving section.

* Solvents used in this procedure must be distilled in glass, suitable for pesticide residue analysis.

* Handle all glassware with clean cotton gloves or precleaned metal tongs.

1) Check ID of sample cartridge match ID on work traveller sheets, and that 2 sets of prelabelled self adhesive labels are attached. These are for identification of sample vials only. **DO NOT USE GUMMED LABELS OR TAPES FOR ANY ANALYTICAL GLASSWARE!**

2) Carefully remove the "Teflon" plug on the bottom of the cartridge. Gently tap the base of the cartridge to ensure that no voids are present in the "Florisil" bed. Remove the screw cap, remove the glass foot from the cap and replace the cap. Place a cooled elution reservoir into the cap, and insert to the level 1.0 cm above the glass wool. Tighten the screw cap to seal, and set in the rack with a watch glass cover over the reservoir.

3) Rinse the tips of each sample tube into a waste beaker with acetone, then pentane to eliminate any external contamination.

4) Set "Kuderna-Danish" 500 ml flask with 10 ml reservoir below, add a precleaned boiling stone to each reservoir, note sample cartridge number and reservoir number.

5) Using one clean 250 ml graduated cylinder , for each lot of sample cartridges dispense 200 ml of pentane into each elution reservoir, covering each reservoir with its watch glass cover. Loosen the plastic cap so the solvent rises 0.5 cm above the

tip of the reservoir and retighten. Ensure that the solvent level in the cartridge tube does not rise continuously upwards. If it does, this indicates a leak in cartridge screw cap seal, and this should be tightened immediately, preventing further rise of the solvent. Pentane solvent reaching the seal may contaminate the sample, and the seal must be replaced! Note discrepancies if they should occur.

6) After elution rinse the tips of each sampling cartridge with 5 ml pentane into the receiver. Remove the "Kuderna-Danish" receiver, assemble the 3 ball "Snyder" column in place, and place 5.0 ml of pentane into the top of the assembled apparatus to wet the column. Proceed with evaporation of the solvent in a temperature controlled water bath (60°C).

7) Continue evaporation of pentane until approximately 1.0 ml of pentane remains in the tip of the reservoir (DO NOT PERMIT THE SAMPLE TO GO DRY - IF THIS DOES OCCUR FOR WHATEVER REASON THIS DISCREPANCY MUST BE REPORTED). Lift the evaporator out of the bath and permit it to cool, until all solvent has drained into the receiver.

Rinse the "Snyder" column with 2.0 ml pentane and remove after it has drained. Rinse the 500 ml flask with pentane sufficient to wet the surface. Do not permit the volume in the bottom reservoir to exceed 10 ml. Remove the 500 ml flask and replace with a micro "Snyder" column. Continue evaporation of the pentane solvent in a tube heater or water bath (60°C), until reduced in volume to 0.5 ml (AGAIN, DO NOT PERMIT THE SAMPLE TO GO DRY, REPORT DISCREPANCIES). Allow samples to cool and rinse the micro "Snyder" column with 0.5 ml pentane. Sample is now ready for "Florisil" clean-up.

8) "Florisil" Column Clean-up (uses precleaned, proven 100/200 mesh "Florisil")

Set up a rack of 15 elution columns with integral solvent reservoirs. Assemble the chromatographic columns by inserting a 0.5 glass wool plug into the bottom using a long glass rod.

"Florisil" will be stored in suitably sized bottles sufficient for the days work (see pg.). Pour "Florisil" from bottle into the empty columns while tapping the column to form a bed of 22 cm (place a 50 ml graduated collection tube under each column). Pack elution columns in groups of 3 or 4 and immediately apply the 1 ml sample to the top of the column using a "Pasteur" pipette with rubber bulb. Rinse the pipette with pentane prior to application of the sample onto the column.

Allow sample to drain onto column just till it drops to the top of "Florisil". Rinse the sample tube with 2 x ½ ml pentane and apply these to the column one at a time as above.

Add pentane 1½ ml at a time until entire column is wetted.

Using one 50 ml graduated cylinder add 40 ml pentane to each reservoir. Collect the pentane eluate in the 50 ml graduated collection tube to a total volume of 14 ml.

9) Place the same micro "Snyder" head previously used for each given sample on the 50 ml graduated collection tube, and proceed with the evaporation of the solvent (without boiling) in a warm water bath until the volume is approximately 5.0 ml. Allow to cool to room temperature. Rinse the micro "Snyder" column down with small portions of pentane.

10) Bring the final volume to 10 ml (accurate) with pentane using a clean "Pasteur" pipette. Cap and swirl gently to ensure a

uniform mix. Using a clean volumetric pipette transfer 5.0 ml of the sample to a 10.0 ml glass ampoule and immediately place in a dry ice/isopropanol bath (DO NOT BLOW ANY SAMPLE TO WASTE) (see pg. 11). The pipette used is rinsed internally and externally with 1.0 and 0.5 ml of pentane respectively into a 10 ml "Kontes" receiver, the same one previously used for this sample. The remaining 5.0 ml of sample is quantitatively transferred to the same 10 ml "Kontes" receiver and the 50 ml graduated collection tube is rinsed with 2 x 1.0 ml portions of pentane and finally 1.0 ml of iso-octane rinse. The sample is gently evaporated to 1.0 ml in a water bath utilizing the same micro "Snyder" head previously used for that sample and sealed in a 1 ml glass - ampoule. (see 12).

11) Glass ampoules are flame sealed after the sample has been thoroughly chilled in a coolant bath.

Either a dry ice/isopropanol bath or a chilled isopropanol bath (utilizing a cryo cooler) may be used.

An appropriate sized piece of plastic "Tygon" tubing is slipped over the neck of the ampoule to permit ease of handling. The ampoule is dipped into the coolant bath for at least one minute to ensure that the sample is thoroughly chilled. After chilling the sample is removed from the bath; the large end of the ampoule slipped into an appropriately sized piece of plastic tubing to permit handling of the ampoule which is immediately sealed using a pin-point flame.

The sealing torch should be positioned so that the flame is horizontal and the ampoule can be held vertically and rotated easily while the sealing is in progress. Ensure that a sturdy tip is produced on the ampoule which will not easily be broken.

While sealing the small ampoules it is necessary to be aware that no PCB's are lost nor that solvent is allowed to evaporate so that the concentration as well as composition is not altered during the process.

12) Samples concentrated to 1.0 ml are transferred without any rinsing using a "Pasteur" pipette to a 1.0 ml glass ampoule which are sealed as in step 11 and appropriately identified with labels provided and submitted for subsequent GC analysis.

NOTE: Unless otherwise indicated by your shift supervisor, all samples will be treated as above. There may be times where you will be requested to put the final extract in a septum capped vial for immediate analysis.

13) Recovery standards will be run along with the first samples to ensure accurate characterization of the PCB's recovery in the clean-up.

VII. INSTRUMENTAL ANALYSIS - DIALY & WEEKLY CHECKS AND OPERATING PROCEDURES

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1) PRIORITIZATION

For the PCB Quantitation step by GC, the following list of priorities will be followed for the purpose of maximizing the overall throughout of samples in this project:

i) Daily Instrument checks as indicated in the Quality Control Instructions. These will include standing current, listing of the analytical file, measurement of noise parameter, and running of the Level B standard.

ii) Glassware rinsing concentrates and split check recoveries so that preparative work can proceed without delay.

iii) Standard Recovery Cartridge (QC) - will be analyzed promptly and results will be reported to MOE personnel on a daily basis.

iv) Weekly Instrument checks as indicated in the Quality Control Instructions. These will include linearity check for each channel and precision checks for each channel.

v) Processed PCB extract samples - to be done in their order of preparation unless otherwise indicated by the technical director.

2) INJECTION TECHNIQUE

Samples will be injected using a standard 10 microlitre syringe for the purpose (e.g. a Hamilton 701N). Both auto injector and manual injection will be used during the course of this project according to the following considerations:

i) Auto injector: Standard operating procedures as indicated in the instrument manual will be used. The injector should be set up to make only 5 microlitre injections, and should use the minimum amount of sample for washing and filling the syringe that is reliable. Remember only 1.0 ml of sample is available; there may be repeat analyses and two other laboratories will be analyzing these extracts.

When using the auto injector the proper information must be filled out on the "Rack and Vial Encoding Sheet" to identify the chromatograms and integrator tapes after analysis is complete.

ii) Manual Injection - An injection volume of 5.0 microlitre will be used and the solvent flush technique is required.

This technique involves drawing 2.0 microlitres of solvent (iso-octane), 2.0 microlitres of air, then a 5.0 microlitres aliquot of sample into the syringe.

Draw the sample volume into the graduated barrel to measure the volume to be injected. If this volume starts to vary significantly from the amount the plunger has withdrawn, first check that the syringe is not being filled too rapidly. If this does not help, the syringe is worn and should be replaced.

This total "package" is manually injected on column for analyses. A minimum of 10 flushings with solvent (iso-octane) is required between sample injections to clean the syringe.

3) OVERNIGHT GC STANDBY CONDITIONS

At the end of the working day, the column oven will be cooled to ambient temperature. The septum will be replaced from a stock conditioned and maintained at elevated temperature. The column oven will be maintained overnight at the maximum temperature used in analysis unless operating an autoinjector.

4) REPORTING OF RESULTS

All results will be tabulated on the "Calculation Summary Sheet". Refer to the "Detailed Procedure for Calculation of Analytical Results" for relevant instructions.

VIII. DETAILED PROCEDURES FOR CALCULATION OF ANALYTICAL RESULTS

GENERAL COMMENTS

1) All analyses and calibrations will be based on a 5 microlitre "on column" injection. Autosamplers will be set up to inject the volumme. Manual injection will incorporate the use of the solvent flush technique as per detailed instructions.

2) Results of analysis will be tabulated on the sheets provided, i.e. "Calculation summary Sheet". Duplicate analysis will be reported on a second sheet.

SPECIFIC DETAILS

1) The calculation files in the integrators used for the analysis will be set up to express results in concentration units (ng/ml).

2) The daily standarization run will be used to generate calibration factors which will be used for that day for all analyses. Refer to detailed procedures in the operational manual of the instrument for details of accomplishing this task. The following basic formula will apply:

$$\text{Cal Fact(i)} = \frac{\text{Conc(i)} \times \text{Scalar}}{\text{Area(i)}}$$

3) Peaks will be identified by their retention times relative to the retention times of the isomers in the Level B standard. A two percent retention window will be used for the purpose of matching peaks.

If a peak does not match any peak in the standard, but does match a known PCB isomer by relative retention time to an isomer in the standard mixture (within two percent), that peak will be considered to be a PCB isomer, and its concentration determined using the calibration factor for the nearest isomer peak in the standard.

4) Concentrations in the extract will be calculated using the calibration factors determined in step 2 for the peaks as identified by step 3. The following basic formula will apply:

$$\mathrm{Conc(i)} = \frac{\mathrm{Cal.\ Fact(i)} \times \mathrm{Area(i)}}{\mathrm{Scalar}}$$

5) Any significantly sized peak which cannot be confirmed by retention time as a PCB isomer will be subjet to confirmation on a second column or by GC/MS, and the concentration calculated as in step 4.

6) Calculation of Corrected Ambient Air Concentrations:
Results determined from step 4 will be converted to give atmospheric concentrations of PCB in ng/m^3, and subsequently corrected for variations in sampling time, temperature and volume recorded during the course of sampling. The following factors must be applied to the concentration analyses for determining the final result:

F = 2 - correction factor for sample split

V_{ext} = final volume of the sample extract in ml

V_{air} = volume of the air sample in m^3 or if reported in ft^3, then $\frac{ft^3}{35.147}$ at the temperature recorded at the sampler's dry grass meter

T_{air} = average temperature of the air sample, as recorded in the sampler's dry gas meter in ^oC, converted to

$$^oK = 273.1 + {^oC}$$

if temperature measured in ^oF, then

$$^oK = 273.1 + 0.56\ (^oF-32)$$

The final calculation of the total PCB in ng/m^3 of air at 25°C (75°F) will be

$$\text{Total} \quad \text{PCB } ng/m^3 = \text{conc (i)} \frac{F.\ V_{ext}.\ T_{air}}{298.1\ V_{air}}$$

PCB PROJECT - QUALITY CONTROL PROGRAM

I. Introduction

II. General Description

III. Quality Control - Specifics

  1) Comprehensive Paperwork/Reporting System
     - Definition and Purpose of Established Forms

  2) Analytical Checks

  3) Instrument Checks

IV. Monitoring of Quality Control Results - "Quality Assurance"

  1) Internal Controls

  2) External Audit

V. Release of Samples for Analysis

VI. Calibration Standards

VII. Instrumental Analysis

I. INTRODUCTION

An overall system of "Quality Control" has been organized to ensure that the objectives of the analytical project are successfully met. This system will provide the necessary data to support the analytical results and will serve to indicate the ongoing validity of the procedures and the final data.

In addition, a system of "Quality Assurance" consisting of both an internal audit and an independant external audit will effectively monitor the total analytical program.

II. GENERAL DESCRIPTION

The quality control program has been divided into three basic areas whereby all aspects of the project are controlled. A detailed list entitled "Definition of Responsibilities" indicates to all project personnel involved, lines of authority, lines of reporting, and all responsibilities and task assignments.

1) Comprehensive Paperwork Reporting System

Detailed forms have been drawn up to present consistant format for logging all information resulting from this project, including analytical data and quality control data. All QC data will be handed to the technical director each day in order to maintain a constant check on all parameters.

All forms relating to this project will be kept in binders in chronological order so that immediate cross-referencing is feasible.

2) Analytical Checks

A series of analytical checks with well defined acceptance criteria has been instituted to monitor analytical levels prior to sampling or prior to sample extraction to ensure that background levels of contamination do not interfere significantly with the analysis.

Also, certain analytical parameters will undergo trend plotting to identify any significant deviations from the norm and to ensure that long term control has been maintained.

3) Instrument Checks

Finally, a series of Instrumental Checks has been developed to monitor the performance of the instrumentation being used for the analyses.

III. QUALITY CONTROL - SPECIFIC DETAILS

1) REPORTING & MAINTENANCE OF INFORMATION FILES

All instrumental checks, analytical checks, and calculations will be tabulated on the forms as detailed below. Samples of each form are provided as an appendix to this procedure.

Each type of form will be kept in a separate three ring binder and maintained in either pure chronological order by date (chronological), or chronological for each site (site) depending on the nature of the information.

All raw data will be filled chronologically by site after all internal and external audits of the data have been performed, and will subsequently be made available to the MOE.

Definition and Purpose of Established Forms

i) "Field Sampling Data Sheet" - (A 45) a record of all parameters relating to the operation of the air sampler in the field with a record of any significant events and/or discrepancies which may be significant to the interpretation of the results. (SITE)

ii) "Daily Summary Sheet" - (A 46) a record which identifies the samples analyzed on any given day, the Glassware Proving numbers for each batch and the analysts who performed the analysis. (CHRONOLOGICAL)

iii) "Work Traveller Sheet" - (A 47) a record sheet for each sample which indicates any exceptions to information on the "Daily Summary Sheet". The actual field identification tag is stapled in place here. The date(s) when the sample was analyzed by GC is also indicated here. (SITE)

iv) "Discrepancy Report" - (A 48) a record sheet for each sample which indicates any deviation from the written or planned procedures. These will be initialled by the chemist in charge. If no comments are present it is assumed that there were no analytical problems with the sample. All sheets must be initialled and present for every sample. (SITE)

v) "Calibration Summary Sheet" - (A 49) a daily record sheet used for each analytical channel to summarize the results of the daily standardization, where parameters such as retention times, response factors, noise levels and standing currents are listed. (CHRONOLOGICAL)

vi) "Detector Precision Summary" - (A 50) a record sheet for tabulating the results of the weekly precision check and the calculated mean, standard deviation and variance for each isomer peak. (CHRONOLOGICAL)

viii) "Rack and Vial Encoding Sheet" - (A 51) a summary sheet for summarizing the processed samples placed into the autosampler for analysis, i.e. a summary of sample numbers, wash vials and standard vials. (CHRONOLOGICAL)

ix) "Standard Composition Summary Sheet" - (A 52) a sheet which summarizes the composition of standards which are prepared and used. The source of the material is indicated (by weighing or dilution) and the concentration of each component of the standard solution. (CHRONOLOGICAL)

x) "Trend Analysis Summary Sheet" - (A 53) a sheet summarizing in numerical form those parameters which are plotted for trend analysis. One sheet may be used for up to four parameters being monitored. (CHRONOLOGICAL)

2) ANALYTICAL CHECKS

The following analytical checks have been set up and will occur daily. These samples will be analyzed following the completion of the daily instrumental checks, and in the following order:

i) Glassware Batch Proving - a batch of glassware corresponding to all glassware, necessary for analysis of a batch of samples will be proven as per detailed instructions. The final extract must have an analysis for total PCB's which does not exceed 1.0 ng/m^3 per set of glassware used for one sample. This calculation will be based on an assumed 10m^3 air sample. Report results on a "Calculation Summary Sheet".

ii) Standard Cartridge Recovery - One sampling cartridge spiked with a mixed isomer standard will be provided by MOE on a daily basis. The cartridge will be processed along with the first batch of samples prepared during the day, and results reported to MOE personnel as soon as available. Calculations will be based on an assumed 10m^3 air sample. Report results on a "Calculation Summary Sheet".

iii) Analytical Blank - Two hundred (200) mls of pentane will be treated as a sample cartridge extract and accompany the first batch of samples which are prepared during the day. Calculations will be based on an assumed 10m^3 air sample. Report results on a "Calculation Summary Sheet".

iv) Field Blanks - A minimum of two field blank cartridges will be extracted and analyzed each day along with the exposed samples. Calculations will be based on an assumed 10m^3 air sample. Report the result on a "Calculation Summary Sheet".

The following analytical checks are also performed but on an "as required" basis:

v) Cartridge Provings - Sampling cartridges are prepared as per detailed instructions. The proving assay which determines whether the cartridge batch is accepted for future use in a survey must not exceed 1.0 ng/m^3 total PCB. The calculation will be based on an assumed 10m^3 air sample. Each batch of 10 cartridges will be assigned a number, CP-#, corresponding to its sequential cartridge proving order. Report results on the "Calculations Summary Sheet".

vi) Cartridge Pre-Shipment Blanks - Prior to shipment of cartridges to the field, blanks must be analysed. Each cartridge batch will have one tube removed and analyzed as if it were a field sample. The analysis must not exceed 1.0 ng/m^3, based on an assumed 10m^3 air sample. Another batch should be substituted if this level is exceeded. The analysis will be assigned a number, CB-#, corresponding to the batch No. Report the results on the "Calculations Summary sheet".

3) INSTRUMENTAL CHECKS

A series of routine checks will be performed to monitor the performance of the GC's. The following checks are to be carried out on a scheduled basis as indicated. Daily checks should occur prior to beginning analysis of samples or after any period in which the instrument was turned off for any reason whatsoever.

Weekly checks should occur on the first working day of the week, usually Monday, and will be concurrent with the necessary daily checks, or after any period in which the instrument was turned off for any reason whatsoever.

Daily Checks

i) Monitor the standing current in each analytical channel used at the initial operating condition prior to sample analysis. Report on the "Daily Calibration Summary Sheet".

ii) List the analytical file, and confirm its validity by comparison to a master record. Attach to "Daily Calibration Summary Sheet".

iii) Measure the noise parameter as indicated by the integrator system. Report on the "Daily Calibration Summary Sheet".

iv) Standardize each channel of the gas chromatograph with one injection of a multicomponent standard at a total PCB level of 500 ng/ml. (Level B). Report the results on the "Daily Calibration Summary Sheet". The attenuation setting used must be selected so that all peaks remain on scale; in the event that analyses by peak height is required. This standard inspection serves the dual purpose of recalibration of the instrumental calculation parameters and as a monitor of the system performance.
Refer to "Detailed Procedure for Calculation of Analytical Results".

Weekly Checks

v) Linearity of each channel will be monitored by one injection of each of three standards having the following total levels of PCB.

Level A = 100 ng/ml
Level B = 500 ng/ml
Level C = 1000 ng/ml

Report results on a "Detector Linearity Summary Sheet" (A 52). Additional standard levels may be added to the linearity check if deemed necessary.

vi) Precision of analyses of each channel will be assessed by an additional four injections of the Level B standard. Report the results on a "Detector Precision Summary Sheet". Utilize Level A and Level B standard on alternate weeks for this test.

IV. MONITORING OF QUALITY CONTROL RESULTS

1) INTERNAL CONTROLS

All instrumental and analytical check results will be tabulated on the appropriate forms provided to the person responsible as soon as they are available on a daily basis.

Trend plotting will be used to monitor the following parameters:

i) retention times of all isomers in the standard
ii) linearity of detector - slope
 - intercept
 - correlation coefficient
iii) standing current in the election capture detector cell
iv) noise parameter as indicated by the integrator
v) glassware provings - total PCB result
vi) analytical blanks - total PCB result
vii) standard recovery - total PCB result
viii) cartridge proving - total PCB result
ix) cartridge blank - total PCB result

These plots will be visually assessed to monitor the results to ensure that the methodology is under control. Where appropriate, acceptable control limits will be established based on statistical considerations.

All samples showing an indicated result above 20 ng/m^3

will have the calculated results rechecked and the peak identifications reconfirmed.

2) EXTERNAL AUDIT BY IEC LIMITED

At least 10% of all samples analyzed will be rechecked and peak identities reconfirmed by an external consultant. This will include rechecking of standardization of the instrumentation and determination of precision and linearity from the raw data outputs supplied.

Priority will be given to samples exhibiting high levels of total PCB or where major peaks are present but are indicated not to be PCB. Analytical data will be delivered weekly for independent audit, and will be returned within the week together with a brief audit report. The external consultants will be allowed full access to the data and will have discretionary authority over the direction of their efforts beyond the basics indicated above. If more appropriate, the weekly external audit will be performed on site at the analytical facility.

V. RELEASE OF SAMPLES FOR ANALYSIS

1) FORMS REQUIRED

Samples will be released on a daily basis for analysis in numbers which can be handled in one day. Samples will be accompanied by a binder containing the necessary record sheets.

- i) "Daily Summary Sheet" one per day
- ii) "Work Traveller Sheet" one per sample
- iii) "Discrepancy Report" one per sample

2) Samples will be released in groupings such that all

sites sampled on any given day are extracted and prepared for analysis together.

Samples should not be prepared for analysis in batches which would necessarily jeopardize a significant percentage of samples from one site in the event of inadvertant contamination or mishap.

3) Samples will be analyzed as per detailed instructions. Changes to these procedures must be received in written form from the Technical Director before any operational procedure is modified.

VI. CALIBRATION STANDARDS

The calibration mixture used will consist of one analytical mixture of pure PCB isomers in iso-octane. Arochlors or other commercially available products will not be used as quantitative analytical standards.

The isomers which are selected for use will be selected based on the following criteria in order,

i) priority should be given to all isomers which are present in commercial Arochlor to an extent greater than 1% on a molar basis.

ii) as many isomers as is feasible will be used, as long as they are baseline resolved or merged to an extent no greater than 20% of the smaller peak.

iii) if two or more isomers are not resolved the isomer which has the greater abundance in commercial Arochlors will be used.

iv) if two or more isomers are not resolved and are of equal abundance, the isomer exhibiting the least detection sensitivity will be used.

The concentration of the stock solution will be about 100 ug/ml Total PCB. The stock solution will be diluted to three levels:

Level A - 1000 ng/ml TOTAL PCB
Level B - 500 ng/ml TOTAL PCB
Level C - 100 ng/ml TOTAL PCB

In all cases, each isomer peak must be detectable and correctly integrated at the lowest level, and in the same mode at all levels.

VII. INSTRUMENTAL ANALYSES

Analysis of all sample extracts, glassware provings, etc., will be performed in the same manner and calculated as per detailed instructions. (See "Detailed Procedure for Calculation of Analytical Results").

Results of the analyses will be tabulated on the "Calculation Summary Sheet" and correctly identified. The following information is considered essential to adequately identify each chromatogram and integrator output tape:

i) Date of Analyses
ii) Sample Number
iii) Attenuation and Range Setting (chart record only)
iv) analysts' Initials

The chromatogram and integrator tape will be stapled together for systematic filing.

CALCULTIONS SUMMARY SHEET

Analyst __________

Date of Analysis __________

Sample No __________

Page No _____ of _____ Pages

Recovery Check __________

Split Check __________

Sample Analysis __________

R (min)	Pk. No.	Isomer(s)	Conc ng/ml	% rec	F_1	F_2 ml	F_3 m^3	F_4	F_5	F6	Conc. ng/m^3	Comments

PCB SAMPLING DATA SHEET

Air Resources Branch
Ontario Ministry of the Environment

Section 1 (To Be Filled By Operator) Nutech Sampler No.: from Samp

Sample No.: THU-S-U1-5 Cartridge Proving (CP) No.: CP-13

Flow Rate: from dial LPM Vacuum Gauge (in. Hg): from dial

Volume Meter	Sampling Time	Temperature
Final Reading From Nutech	Time OFF 8:15 PM	Time OFF 18°C
Initial Reading From NuTech	Time ON 8:00 PM	Time ON 20°C
Volume Sampled Difference cf ☐ m³ ☒	Time (hrs) 24.25 hr	

Please ignore spelling error

Dat~~a~~e Cartridge Received	Dat~~a~~e Cartridge Exposed	Date Shipped for Analysis
SEPT 17, 1979	START - 20/9/79 END - 21/9/79	SEPT 23, 1979.

General Weather Report for Sampling Day (Indicate by X)

Day Time: ✓ Sunny ___ Cloudy ___ Stormy ___ Rain/Snow ✓ Windy ___ Still

Night Time: ___ Cloudy ___ Stormy ___ Windy ✓ [✓ Rain]/Snow

Remarks (Use Other Side If Necessary)

① could smell local industrial plant (specify)
② teflon seal seemed loose when replaced after sampling.

Section II (To Be Filled By Contractor) Sample No.: ______

Blank Cartridge Data

Date Prepared	Blank Test Date	Date Shipped for Sampling	Approving Officer

Sample Data

Date Received	Date Extracted	Date Analyzed	PCB/ Extract	"Average" Wind Speed/ Direction

Remarks (Use Other Side If Necessary)

PCB - ANALYSIS SHEET

"DAILY SUMMARY"

Date ____________ **Analyst** **Extractions** ______ **initial**

Clean Ups ______

Sample No. **Batch 1** ____________ **to** ____________ **inclusive**

Batch 2 ____________ **to** ____________ **inclusive**

Batch 3 ____________ **to** ____________ **inclusive**

Glassware Proving No. for: **Batch 1** ____________

Batch 2 ____________

Batch 3 ____________

Solvent Lot No.	C_5	i - C_8
Batch 1		
Batch 2		
Batch 3		

Florisil Clean Up - Split Volume ______ **ml** ______ **(initial)**

*** Note:** all samples extracted have been split into two fractions after clean-up which have been sealed into glass ampoules for future analysis and labelled.

Glassware has been rinsed with appropriate solvents and placed in the oven at 250°C for the next days samples.

SIGNATURE ____________________

PCB SAMPLE ANALYSIS SUMMARY SHEET - "WORK TRAVELLER"

STAPLE
SAMPLE
IDENTIFICATION
TAG HERE

SAMPLE NO. ____________

BLANK NO. ____________

DATE OF RECEIPT ____________

Date of Sampling ____________

Date of Extraction & Clean Up ____________

Date of Analysis	____________	Analyst	____________	Inj. Vol.	________
	____________		____________		________
	____________		____________		________

Instrument - Varian 3700 ___ Channel A ___
Channel B ___

HP 5709A ___
HP 5880 ___ Channel A ___

* Glassware Proving Batch No. ____________

* Solvent Lot No. ____________

* Split Volume ____________ ml

Discrepancies - please report any deviations from written procedure on the page following entitled "Discrepancy Report"

* Indicate only if different from that which is reported on the "Daily Summary Sheet"

DISCREPANCY REPORT

NO.	Sample No. Sample Preparation as per instruction Yes No if no please indicate discrepancies below.	Initial

DAILY PCB CALIBRATION SUMMARY

INSTRUMENT ______________ CHANNEL ________ INJ. VOL. ______

DATE ___________ STANDING CURRENT at T°C INITIAL ____________

OPERATOR ______________

STANDARD SOLUTION ID ____________________ DATE OF PREPARATION ______________

PEAK NO.	RET. TIME	CAL. FACTOR	PEAK NO.	RET. TIME	CAL. FACTOR
1			29		
2			30		
3			31		
4			32		
5			33		
6			34		
7			35		
8			36		
9			37		
10			38		
11			39		
12			40		
13			41		
14			42		
15			43		
16			44		
17			45		
18			46		
19			47		
20			48		
21			49		
22			50		
23			51		
24			52		
25			53		
26			54		
27			55		
28			56		

INCLUDE CHROMATOGRAMS AND RAW DATA WITH SUMMARY SHEETS (USE ONE FOR EACH STANDARD RUN)

DETECTOR PRECISION SUMMARY

Analyst ________

Date __________ Instrument __________ Channel _________

Standard _________ Page No. _____ of ______

Pk No.	Run 1		Run 2		Run 3		Run 4		Mean		% S_D	
	R_T	Area	R_T	Area	R_T	Area	R_T	Area	R_T	Area	R_T	Area

RACK & VIAL ENCODING SHEET

Date of Analysis ______________ Inj Vol Setting #1 __________

Analyst ______________ Inj Vol Setting #2 __________

Rack No. No. inj/vial Inj. Volume					
Vial No.					
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					

PCB STANDARD COMPOSITION SUMMARY SHEET

Date of Preparation ________________ Primary Std _______

Analyst _________ Secondary Std _____

Std No. _________________ Final Volume _________

Page No. ________ of __________

No.	Isomer	Initial Concentration	Volume Sampled	Dilution Factor	Final Concentration

TREND ANALYSIS SUMMARY SHEET

Parameter					
Units					
Date					

DETECTOR LINEARITY SUMMARY

Analyst __________ Page No. _____ of ______ Level A Std # ______________

Date __________ Level B Std # ______________

Instrument _______ Channel _________ Level C Std # ______________

Pk	Integrator Area Counts			Linear Regression y=a+bx		
No	Std A	Std B	Std C	a	b	r^2
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						

OPERATING INSTRUCTIONS FOR THE NUTECH SAMPLER

Initial Setup of Sampler:

(1) Remove front panel of the sampler.

(2) Connect the 120 volt power cord to the sampler and 110V, 60HZ, AC outlet, regular Ontario Hydro outlet. On the left side of the control panel (see figure) flip the D.C. switch up. Nothing should happen. Flip the A.C. switch up, the motor and pump will go on. Flip the A.C. switch down and the pump and motor will go off. Keep the D.C. switch in the upward position at all times. If the machine does not go on, check that the power cord has been plugged firmly into the machine and the outlet. Try again. If the machine does not go on, call Dr. Naz Hija-i in Toronto (416-965-4081).

(3) Insert the provided attachment from the Quick Connect side (see figure) to the machine panel labelled inlet. Push the attachment in until you hear the distinct snap. Pull on the attachment; if it does not depart from the panel, then the attachment is securely in place.

(4) Rotate the attachment so that the Knurled nut part is in a vertical position. Push the copper tube into the Cupboard Catcher. The attachment in this position should only sway a fraction of an inch either way. If not, tighten the Cupboard Catcher by pushing it between your thumb and forefinger while the copper tube is in position.

(5) On the right hand side of the sampler panel, there are three holes and two stove fly bolts. Unscrew the stove bolts all the way out. Position the provided aluminum measuring cup (rain cover) so that the clearnance between the knurled nut and the measuring cup is about 10 centimeters. Attach the measuring

cup arm by screwing the stove bolts through the arm and the appropriate two holes in the panel. This must be very firm to withstand strong winds. Bend the copper tube and/or the arm so that the Knurled Nut is approximately facing the middle of the measuring cup. Now you are ready to install the sampling cartridge.

Sampling Cartridge installation:

READ THE SAMPLING CARTRIDGE HANDLING PROCEDURE (separate sheet).

(1) Loosen the Knurled Nut by turning counter clockwise (half a turn only). Insert the cartridge and re-tighten by turning the Knurled Nut clockwise. No tools are needed, finger tightening is sufficient. Pull the cartridge slightly to make sure it is properly and firmly seated. If the cartridge comes out, finger tighten the Knurled Nut until the cartridge does not come out of the fitting when pulled out. Do not use excessive force or twisting motion, otherwise the cartridge might break*.

*** If the cartridge breaks for any reason, use the spare cartridge that you should be carrying and note that in the Remarks section of the Data Sheet.**

Sampling Data Sheet:

Initial Readings -

(1) Set the flow control valve to the maximum by turning counter-clockwise. Turn A.C. switch ON (upright position). The motor and pump will go on. Adjust the flow control valve to achieve a reading between 7-10 liters per minute (LPM) on the flow meter. Use the centre of the flow meter float. If the machine cannot achieve 7 LPM when the valve is fully turned counter clockwise, record the actual reading in the appropriate space on the Data Sheet and the time.

(2) Fill in the appropriate readings from the Vacuum Guage, Volume Meter and Temperature Gauge.

(3) Replace the front panel cover and fill in the other information and any remarks on the machine operation, any difficulty or accidental mishandling of the cartridge.

Final Readings -

(1) On the second day, when you arrive for removing the exposed cartridge and installing a fresh one, inspect the sample and positioning of the cartridge for any signs of tampering with the equipment. Please write all necessary notes in the Remarks section.

(2) Remove front panel cover and turn machine OFF (A.C. switch in downward position). Take the readings on the Volume Meter and Temperature Gauge. Note any changes in the readings on the Vacuum Gauge or the Flow Meter. Please make a note of any large differences.

(3) Loosen the Knurled Nut about half a turn and pull the cartridge out. Recap the cartridge as in the Cartridge Handling Procedure (separate sheet).

(4) Insert the fresh cartridge and follow the same procedure as before.

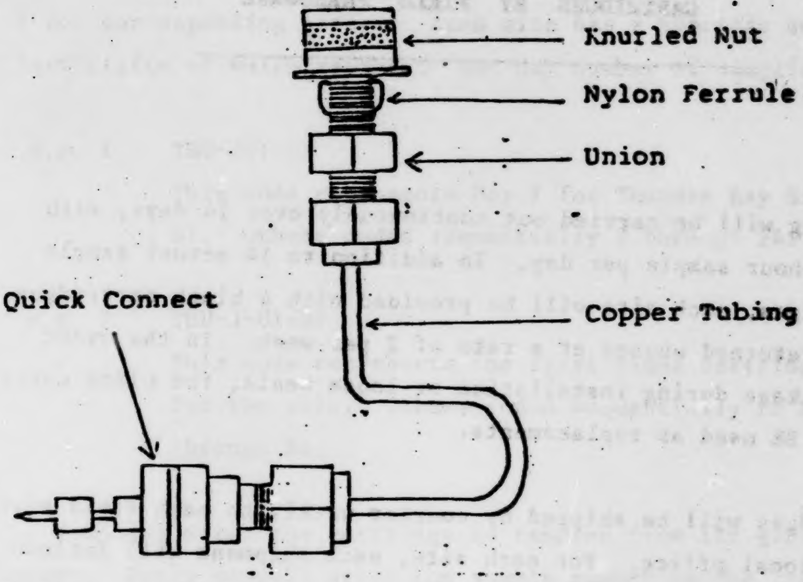
(5) Please follow the instructions supplied regarding handling and shipping of cartridges.

If you have any problems, call your local Ministry of the Environment contact or Dr. Naz Hijazi in Toronto (416-965-4081).

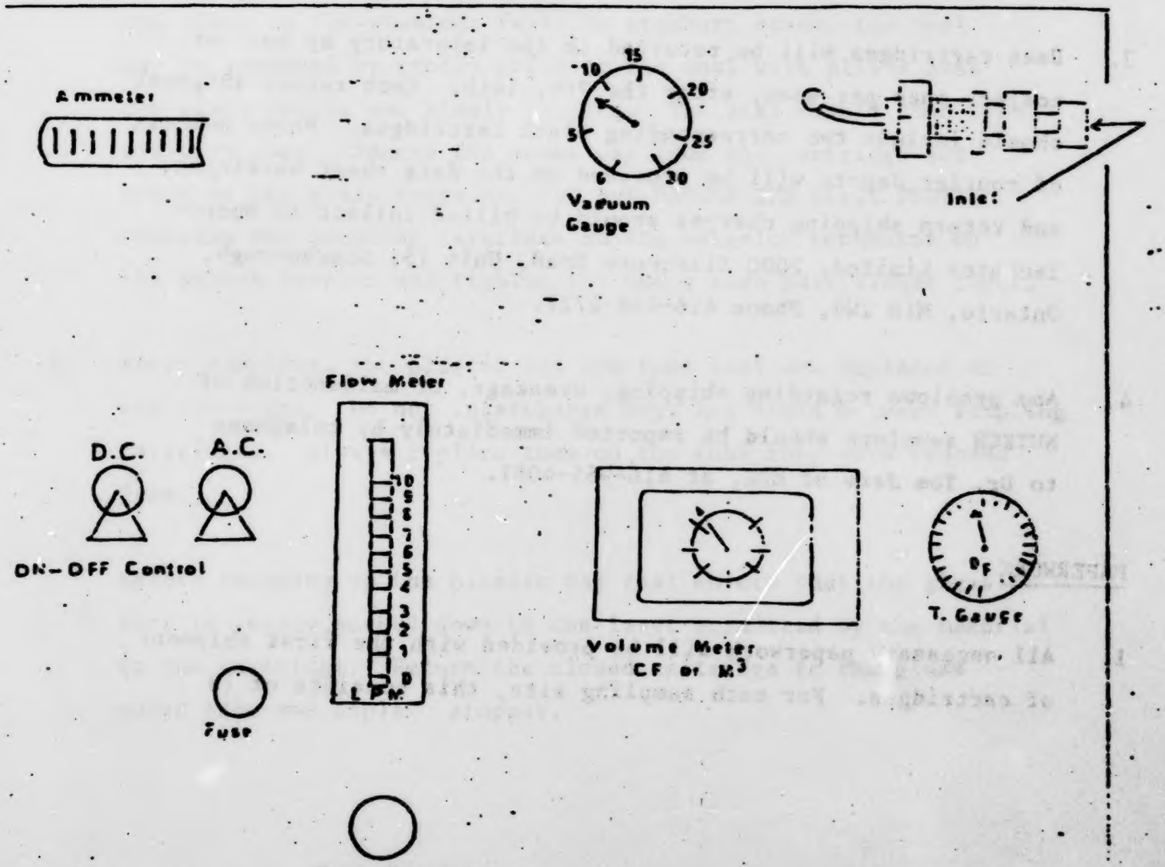
If unavailable, leave a message with the clear instructions to the secretary that it is an emergency call and give your phone number where you could be reached within the next few hours.

****

Thank you for your co-operation.


Knurled Nut
Nylon Ferrule
Union
Quick Connect
Copper Tubing

CONTROL PANEL


Ammeter
10
15
20
5
25
6
30
Vacuum Gauge
Inlet
Flow Meter
10
9
8
7
6
5
4
3
2
1
0
LPM
D.C.
A.C.
ON-OFF Control
Fuse
Volume Meter CF or M³
°F
T. Gauge
Flow Control

INSTRUCTIONS FOR HANDLING OF SAMPLE CARTRIDGES BY FIELD PERSONNEL

General

1. Sampling will be carried out continuously over 14 days, with one 23-hour sample per day. In addition to 14 actual sample cartridges, each site will be provided with 4 blank cartridges to be returned unused at a rate of 2 per week. In the event of breakage during installation or loose seals, the blank cartridge SHOULD BE used as replacements.

2. Cartridges will be shipped by courier weekly to each field site or regional office. For each site, each shipment will include 9 cartridges, 7 to be used as samples and 2 as blanks. Shipping of cartridges will be timed to arrive 1-2 days before the previous supply has been used up.

3. Used cartridges will be returned to the laboratory by courier collect once per week, after the 7th, 14th. Each return shipment should include two corresponding blank cartridges. Phone numbers of courier depots will be provided on the data sheet envelopes, and return shipping charges should be billed collect to Nucro-Technics Limited, 2000 Ellesmere Road, Unit 16, Scarborough, Ontario, M1H 2W4, Phone 416-438-6727.

4. Any problems regarding shipping, breakage, or malfunction of NUTECH samplers should be reported immediately by telephone to Dr. Tom Jarv of MOE, at 416-965-4081.

PAPERWORK

1. All necessary paperwork will be provided with the first shipment of cartridges. For each sampling site, this consists of 18

coded "PCB Sampling Data Sheets", 14 for actual sample cartridges and 4 for corresponding blanks. Each site has a specific code, the last digits of which represent the day number of sampling:

e.g. 1 THU-JU1-1
This code represents Day 1 for Thunder Bay Site U1. Others coded sequentially 2 through 14.

e.g. 2 THU-J-U1-B1
This code represents the first Blank cartridge for the site. Others coded sequentially to B2 through B4.

4. At the sampling site; the cartridge is removed from its glass container. Using pencil, write the sample number on the tag attached to the cartridge. Unwrap the aluminum foil and place on the sampler. Remove the teflon tube seal from the cartridge by gently twisting and pulling (some resistance should be felt) and place on the aluminum foil. In stubborn cases, the seal may be loosened by gently gripping the seal with pliers over the glass tubing and slowly turning. The seal should then come off with ease. Remove the screw cap from the cartridge and place in the glass container (DO NOT HANDLE THE GLASS FOOT). Position the sampling cartridge in the swagelok connector on the Nutech Sampler and tighten the nut ¼ turn past finger tight.

5. After sampling, the plastic cap and tube seal are replaced on the cartridge. Do not interchange caps and seals between sampling cartridges. Always replace them on the tube they were removed from.

Before snugging up the plastic cap seal ensure that the glass foot is gently pushed down to the level permitted by the material in the cartridge. Return the closed cartridge to the glass outer tube and replace stopper.